

# QUALIFICATION OF TRIVALENT CHROMATE AS A HEXAVALENT CHROMATE ALTERNATIVE FOR PROPELLANT AND CARTRIDGE ACTUATED DEVICES

*Harry L. Archer*

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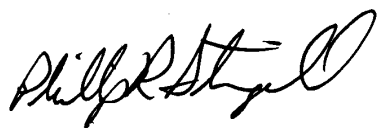
## FOREWORD

The work reported herein was performed at the Indian Head Division, Naval Surface Warfare Center, Indian Head, MD.



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## ACKNOWLEDGMENTS

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## CONTENTS

<i>Heading</i>	<i>Page</i>
Foreword .....	iii
Acknowledgments .....	v
Introduction .....	1
Objective .....	1
Approach .....	5
Test Results and Discussion .....	17
Conclusion.....	29
Recommendations .....	30
References .....	31

## Tables

I. Test Panel Plating Preparation.....	8
II. Hexavalent Chromate Replacement Panel Test Matrix .....	8
III. Plating Preparation for PADs and CADs.....	12
IV. Chip Resistance Test TCP/Zinc-Nickel Results .....	27
V. Unscrew Torque Typical PADs and CADs Test Results <sup>1</sup> .....	28

## Figures

1. Zinc-Nickel Plating, Low-Nickel, Low Embrittlement, Hexavalent-Chromate Free (ID 12)5,6,7,8,9,10,11,12,13 .....	2
2. Zinc-Nickel Plating, High-Nickel, Low Embrittlement, Hexavalent-Chromate Free (ID 12x)5,6,7,8,9,14,12,13 .....	3
3. Trivalent Chromate Conversion Coating.....	4
4. Test Panel Configuration .....	7
5. Typical Adhesive Used to Seal USRM Motor Tubes .....	9
6. PAD MK 74 Manifold Torque Test Item .....	10
7. PAD MK 74 Manifold Tube Torque Test Item .....	11
8. Modified MK 74 to Motor Tube Test Assembly.....	13
9. CAD Torque Test Assembly .....	14
10. Zinc-Nickel Plating, Low-Nickel, Low Embrittlement, Hexavalent-Chromate Free Rework (ID 12)5,11,8,9,10,12,13 .....	15
11. Zinc-Nickel Plating, High-Nickel, Low Embrittlement, Hexavalent-Chromate Free Rework (ID 12x)5,11,8,9,14,12,13.....	16
12. Photos of TCP/Zinc-Nickel/Steel Panels After Various Stages of Salt Fog Testing Results .....	19
13. 1000 Hr Cyclic Salt Fog Test of TCP/Zinc-Nickel/Steel Panels .....	20
14. 2000 and 3000 Hr Cyclic Salt Fog Test of TCP/Zinc-Nickel/Steel Panels .....	21
15. Sulfur Dioxide/Salt Fog Testing on TCP/Zinc-Nickel/Steel Panels After Various Stages .....	22
16. 92 Hr (4 days) Sulfur Dioxide/Salt Fog Testing on TCP/Zinc Nickel .....	23
17. 168 Hr (1 Week) Sulfur Dioxide/Salt Fog Testing on TCP/Zinc Nickel.....	24
18. Painted After Various Stages Of Sulfur Dioxide/Salt Fog Testing Per Table II .....	25
19. Photos of Painted after 1872 Hrs (78 days) Sulfur Dioxide/Salt Fog Testing.....	26



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## INTRODUCTION

On February 28<sup>th</sup>, 2006, the Occupational Safety and Health Administration changed the permissible exposure limit for hexavalent chromate<sup>1,2</sup> *from 52 to 5µgrams per cubic meter*. This change came *after* IHDIV/NSWC qualified a hexavalent chromate conversion coating (CCC) for use on unpainted PADs. This new requirement will make hexavalent chromated products difficult to procure in the near future.

Testing described herein qualifies a non-proprietary trivalent chromate (also known as TCP) (Figure 3) over zinc-phosphate coating system to replace hexavalent chromate on zinc-nickel plated carbon steel PADs and CADs. We previously tested and qualified a zinc-nickel plating system (see IHTR 2694<sup>3</sup>). These include:

- hexavalent chromate on zinc–nickel
- primed/painted hexavalent chromate on zinc–nickel,
- primed/painted TCP over zinc-phosphate on zinc–nickel.

However, IHDIV/NSWC did not previously test unpainted TCP over zinc-phosphate on zinc–nickel. The photo in the bottom of Table VIII in IHTR 2694<sup>3</sup> shows no corrosion, even in the score on the bottom of the painted TCP test panel. Therefore, only limited testing is required to qualify this TCP plating system. Moreover, unpainted TCP weren't previously tested for gravel resistance.

Often CADs have more exposure to marine and shipboard smoke stack sulfur dioxide emissions than PADs, therefore testing for this type environment would be useful.

Plating typically changes the torque retaining power of threads. Adhesives used to lock thread in place often lubricate threads to facilitate assembly. Therefore, it is useful to determine if this new plating adversely affect the holding power of the torqued thread.

## OBJECTIVE

IHDIV/NSWC's objective is to qualify plating (Figure 1) 513-174-0215, *Zinc-Nickel Plating, Low Nickel, Low Embrittlement, Hexavalent Chromate Free* and a higher nickel version (12 percent versus 5 percent) (Figure 2) SK07005-E213K-2 for steel unpainted shipboard/marine propellant actuated devices (PADs), cartridge actuated devices (CADs) environments. We also desire to establish the durability of steel plated in accordance with drawing 513-174-0215 and a higher nickel (12 percent versus 5 percent) version of SK07005-E213K-1 (Figure 2) to withstand shipboard smokestack sulfur/sulfuric acid and salt fog emissions.



CLASSIFICATION OF CHARACTERISTICS (DD-STD-210)		REVISIONS	
CRITICAL-	NONE	REV	DESCRIPTION
MAJOR-	4	DATE	APPROVAL
MINOR-	ALL OTHER CHARACTERISTICS		
<p>NOTE:</p> <ol style="list-style-type: none"> <li>INTERPRET DRAWING IN ACCORDANCE WITH ASME Y14.100-2004 WITH APPENDICES B, C, D, AND E.</li> <li>(M101) 2. BEFORE ANY HEAT TREATING, CLEAN ALL SURFACES IN ACCORDANCE WITH ASTM B 322-99. ALL CLEANING SHALL BE CYANIDE FREE.</li> <li>(M102) 3. AFTER FINAL MACHINING, STRESS RELIEVE IN ACCORDANCE WITH ASTM B 849-94. AFTER STRESS RELIEVING AND BEFORE ANY PLATING, ALL SURFACES SHALL REMAIN HYDROCARBON OIL FREE.</li> <li>4. PROTECT FROM MOISTURE BEFORE DESCALING. DESCALE PER MIL-S-50020 AMENDMENT 2 AND THEN IMMEDIATELY CLEAN PER ASTM B 322-99. DESCALING AND CLEANING SHALL BE CYANIDE FREE.</li> </ol> <p>NON-MANDATORY ADVISORY INFORMATION:</p> <p>A LOW TOXIC RUST REMOVER WAS FOUND AS AN EFFECTIVE CYANIDE FREE DESCALER IN SOME APPLICATIONS. RUST REMOVER GDP 1000 GLOBAL DIVERSIFIED PRODUCTS 11400 47TH ST. N. CLEARWATER FL 33767 (727) 571 3777</p> <ol style="list-style-type: none"> <li>(M103) 5. IMMEDIATELY AFTER NOTE 4 CLEANING, ACTIVATE AND NICKEL-PHOSPHORUS COAT AS PER ASTM B 733-97, TYPE V, CLASS I, SERVICE CONDITION SC0, TO 0.0001 THICKNESS MAXIMUM.</li> <li>(M104) 6. ZINC-NICKEL PLATE IN ACCORDANCE WITH ASTM B841-99, CLASS I, WITH 5% TO 7% NICKEL, BAKE IN ACCORDANCE WITH SAE AMS 2759/9A WITHIN THREE HOURS AFTER PLATING. MAXIMUM BAKING TIME SHALL BE 26 HOURS.</li> <li>7. ZINC PHOSPHATE CONVERSION COAT PER ASTM D2092-95 METHOD A.</li> <li>8. TRIVALENT CHROMATE CONVERSION COAT PER MIL-DTL-81706B, TYPE III, CLASS IA, FORM 1, METHOD C, (M817062A1C) USING DRAWING S13-174-0216 QUALIFIED SOURCE(S).</li> </ol>		<p>CAD MAINTAINED, CHANGES SHALL BE INCORPORATED BY THE DESIGN ACTIVITY</p>	

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**Figure 1. Zinc-Nickel Plating, Low-Nickel, Low Embrittlement, Hexavalent-Chromate Free (ID 12)5,6,7,8,9,10,11,12,13**



<p>NOTE:</p> <ol style="list-style-type: none"> <li>INTERPRET DRAWING IN ACCORDANCE WITH ASME Y14.100-2004 WITH APPENDICES B, C, D, AND E.</li> <li>ONLY ITEMS DESCRIBED ON THIS DRAWING ARE APPROVED FOR USE IN THE APPLICATIONS SPECIFIED HEREON. A SUBSTITUTE ITEM SHALL NOT BE USED WITHOUT PRIOR APPROVAL BY THE QUALIFYING ACTIVITY.</li> <li>IDENTIFICATION OF THE APPROVED SOURCE(S) OF SUPPLY HEREON IS NOT TO BE CONSTRUED AS A GUARANTEE OF PRESENT OR CONTINUED AVAILABILITY AS A SOURCE OF SUPPLY FOR THE ITEM DESCRIBED ON THE DRAWING.</li> </ol>	<p style="text-align: center;">CLASSIFICATION OF CHARACTERISTICS (DOD-STD-2101)</p> <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td>CRITICAL:</td> <td>NONE</td> </tr> <tr> <td>MAJOR:</td> <td>NONE</td> </tr> <tr> <td>MINOR:</td> <td>ALL CHARACTERISTICS</td> </tr> </table>	CRITICAL:	NONE	MAJOR:	NONE	MINOR:	ALL CHARACTERISTICS	<p style="text-align: center;">REVISIONS</p> <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <th>REV</th> <th>DESCRIPTION</th> <th>DATE</th> <th>APPROVAL</th> </tr> <tr> <td> </td> <td> </td> <td> </td> <td> </td> </tr> <tr> <td> </td> <td> </td> <td> </td> <td> </td> </tr> <tr> <td> </td> <td> </td> <td> </td> <td> </td> </tr> </table> <p style="text-align: center;">CAD MAINTAINED. CHANGES SHALL BE INCORPORATED BY THE DESIGN ACTIVITY</p>	REV	DESCRIPTION	DATE	APPROVAL												
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Figure 3. Trivalent Chromate Conversion Coating

## **APPROACH**

### **Test Panel Environmental Testing: Salt Fog and Chip Resistance Testing**

Salt fog and chip resistance testing were both performed at Naval Surface Warfare Center, Carderock Division using the same equipment used to test panels qualified in IHTR 2694<sup>3</sup>. Sulfur dioxide testing was performed at NAVAIR Patuxent River.

#### **Test Article**

Panels were fabricated in accordance with 5130K-CAD-REPL-0100 (see Figure 4). These panels used the same steel roll stock as the ones used in IHTR 2694<sup>3</sup> and the low volatile organic content paint system<sup>4</sup> qualification. Moreover, all these test panels were the same configuration except the unpainted controls, which were larger, flat, and not from this steel stock.

#### **Preparation**

Panels were plated in accordance with Table I.

#### **Testing**

Panels were exposed to Table II cyclic salt fog testing. This is the same cyclic salt fog testing used to qualify the PAD paint system<sup>4</sup>.

### **Torque Testing**

We also compared the torque required to unscrew typical plated PAD and CAD items bonded with Eccobond® 45, Catalyst 15 (Figure 5) to assure that the coating does not significantly contribute to the loss of bonded screwed joint holding torque.

#### **PADs**

##### **Test Article**

There are many types of PAD parts. The Mk 74 underseat rocket motor (USRM) with its many threaded surfaces should be a good representation of threaded PADs.

##### **Preparation**

The Mk 74 is no longer a production item, so the igniter threads were trimmed, shown in Figure 6, to prevent them from reentering production.

The USRM and associated motor tubes (Figure 7), were plated according to Table III and assembled in accordance with Figure 8 with the same plating. All torque test items were from reworked parts. Therefore, they were stripped and plated according to 513-174-0213 (Figure 10) and SK07005-E213K-1 (Figure 11).

### **Testing**

All assemblies were allowed to cure for at least 1 week. Afterwards, the tubes were unscrewed from the bodies with a torque wrench and the torque at first movement was recorded.

### **CADs**

#### **Test Article**

MC 50 initiator steel body with aluminum M720 initiator heads represents typical CADs. This CAD was chosen because the parts were readily available and they have established torque test procedures.

#### **Preparation**

The bodies were plated according to Table III, and assembled according to Figure 9.

All torque test items were from reworked parts. Therefore, they were stripped and plated according to 513-174-0213 (Figure 10) and SK07005-E213K-1 (Figure 11).

### **Testing**

All assemblies were allowed to cure for at least 1 week. Afterwards, the tubes were unscrewed from the bodies with a torque wrench and the torque at first movement was recorded.

### **Compatibility**

As with any new coating, its compatibility must be considered if it is in contact with or outgases to propellant. Samples Mechanite 19 propellant were tested with samples of TCP to assess whether or not the TCP makes the propellant more reactive.





**Table I. Test Panel Plating Preparation**

Item No.	Qty	Preparation Description for Drawing SK5130K-CAD-REPL-0100 (Figure 4)
12	9	1. Mark the number "12" (no quotes) on a corner near the ¼ in hole. 2. Plate panel in accordance with drawing 513-174-0215 (Figure 1).
12x	8	1. Mark the number "12x" (no quotes) on a corner near the ¼ in hole so that the item number can be seen after finishing. 2. Plate panel in accordance with drawing SK07005-E213K-1 (Figure 2).
Control	6	4 ½ in x 6 in 4130 Steel panels without weldment or plating
6p2 <sup>i</sup> Reference only	6	1. Mark the number "6p2" (no quotes) on a corner near the ¼ in hole so that the item number can be seen after finishing. 2. Plate panel in accordance with drawing 513-174-0215. 3. Prime and paint as per MIL-DTL-85097[15]. Primer coating shall be per MIL-P-53030[16]. Paint shall be as per MIL-PRF-85285[17], Color, Gloss White No. 17935 in accordance with FED-STD-595[18].

<sup>i</sup> Note: The 6p2 panels were previously subjected to 10 day humidity per ASTM D2247-99[19], and MIL-P-83126A[20], Section 4.4.2.14.2 and 120 day salt fog per ASTM G85 ANNEX 5[21]. Each panel has been scored diagonally in an "X" pattern on the side opposite the channel. Also, the paint adhesion was tested.

8

**Table II. Hexavalent Chromate Replacement Panel Test Matrix**

Tests	Zinc – Nickel with Trivalent Chromate Conversion			
	Unpainted			Painted (panel ID #6p2 from test in IHTR 2694 pp 20)
Drawing SK5130K-CAD-REPL-0100 Panel Plated Per 513-174-0215 or 513-174-0213 if Reworked				
Rain test <sup>h</sup>	ID# 12, Qty 3	ID# 12x, Qty 3		
Humidity <sup>i</sup> test	Use the same panels used in “h” above	Use the same panels used in “h” above		
Salt fog <sup>j</sup>	Use the same panels used in “i” above	Use the same panels used in “i” above		
Chip resistance <sup>k</sup>		ID# 12, Qty 3	ID# 12x, Qty 3	
Salt fog <sup>n</sup> SO <sub>2</sub>		ID# 12, Qty 3	ID# 12x, Qty 3	ID# 6p2, Qty 6

<sup>h</sup> MIL-P-83126A[20], Section 4.4.2.14.1

<sup>i</sup> ASTM D2247-99[19], and MIL-P-83126A[20], Section 4.4.2.14.2

<sup>j</sup> ASTM G85[21] ANNEX 5, 168hours and 1000hours (2000 & 3000 hours optional)

<sup>k</sup> ASTM D3170-03[22]

<sup>n</sup> ASTM G85[21] Annex A4 Section A4.4.4.1 Salt/SO<sub>2</sub> Spray (Fog) Testing.

CLASSIFICATION OF CHARACTERISTICS (DOD-STD-210)		REVISIONS	
CRITICAL - NONE		REV	DESCRIPTION DATE APPROVAL
MAJOR - NONE		A	SEE ECP 80-5130-203 5/9/81 B.L. Perkins
MINOR - ALL OTHER CHARACTERISTICS		B	REDRAWN WITH CHANGE SEE ECP 01018-5130M 8/7/91 J.L.G.
		C	REDRAWN WITH CHANGE SEE ECP 04020-5130M 4/12/91 J.S.
CAD MAINTAINED. CHANGES SHALL BE INCORPORATED BY THE DESIGN ACTIVITY			

**NOTES:**

- INTERPRET DRAWING IN ACCORDANCE WITH ASME Y14.100-2000 WITH APPENDICES B, C, D, AND E.
- ADHESIVE IS A TWO COMPONENT, CONTROLLED FLEXIBILITY EPOXY MATERIAL FOR USE WHERE SHOCK AND PEEL RESISTANCE ARE DESIRED.
- ADHESION TO GLASS, METALS, CERAMICS AND PLASTICS IS EXCELLENT.
- THE FLEXIBILITY OF ADHESIVE IS DETERMINED BY THE AMOUNT OF CATALYST USED.
- IDENTIFICATION OF THE APPROVED SOURCES OF SUPPLY HEREON IS NOT TO BE CONSTRUED AS A GUARANTEE OF PRESENT OR CONTINUED AVAILABILITY AS A SOURCE OF SUPPLY FOR THE ITEM DESCRIBED ON THIS DRAWING. ONLY THE ITEM DESCRIBED ON THIS DRAWING, WHEN PROCURED FROM THE VENDOR(S) LISTED HEREON, IS APPROVED BY THE NAVAL SURFACE WARFARE CENTER, INDIAN HEAD, MD FOR USE IN THE APPLICATION(S) SPECIFIED HEREON. A SUBSTITUTE ITEM SHALL NOT BE USED WITHOUT PRIOR TESTING AND APPROVAL BY THE NAVAL SURFACE WARFARE CENTER, INDIAN HEAD, MD 20640.

**TYPICAL PROPERTIES (SEMI-RIGID FORMULATION)**

PROPERTIES	VALUE
TEMPERATURE RANGE	-70°F TO 250°F
HARDNESS, SHORE A	70
COLOR	BLACK
BOND STRENGTH IN SHEAR AT ROOM TEMPERATURE, PSI	3100
FLEXURAL STRENGTH, PSI	5500
I200 IMPACT, FT LB/IN OF NOTCH	4.0
DIELECTRIC STRENGTH, VOLTS/MIL	400
DISSIPATION FACTOR, 10 <sup>4</sup> TO 10 <sup>6</sup> Hz	.03 TO .04
MIXTURE: ECCOBOND AND CATALYST	1 TO 1 BY WEIGHT
POT LIFE	3 HOURS MIN AT ROOM TEMPERATURE
CURE TIME-ROOM TEMPERATURE	24 HOURS MINIMUM
	30 MINUTES MINIMUM
	15 MINUTES MINIMUM

APPROVED SOURCE(S) OF SUPPLY		
VENDOR	VENDOR'S ITEM IDENT NO.	APPLICATION
NATIONAL STARCH AND CHEMICAL CO. EMERSON AND CUMING SPECIALTY POLYMERS DIV 869 WASHINGTON ST CANTON, MA 02021-2513 PHONE 781-828-3309 CAGE CODE 04552	ECCOBOND 45 CATALYST 15	EPOXY ADHESIVE USED ON ROCKET MOTORS AND ROCKET CATAPULTS

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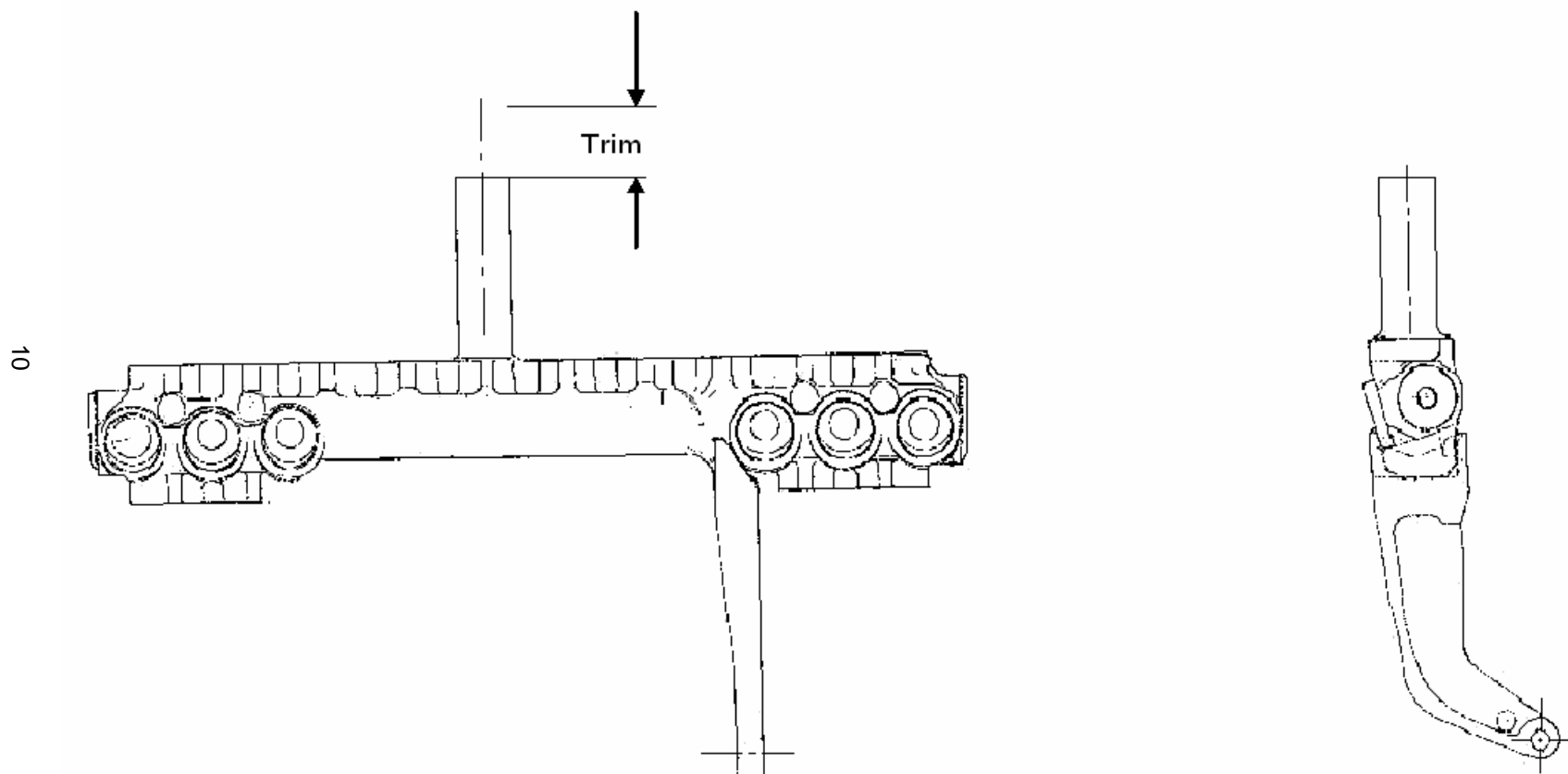
**SOURCE CONTROL DRAWING**

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES		INDIAN HEAD DIVISION NAVAL SURFACE WARFARE CENTER INDIAN HEAD, MD 20640-5035		DEPARTMENT OF THE NAVY NAVAL AIR SYSTEMS COMMAND PATUXENT RIVER, MD 20670-1628	
GENERAL TOLERANCES		ENGINEER		ADHESIVE, EPOXY RESIN	
XX DECIMALS ± —		ENGINEER			
XXX DECIMALS ± —		CHECKED			
ANGLES ± —		DRAWN			
MATERIAL:		APPROVED FOR MFG	DATE	SIZE	CAGE CODE
NEXT ASSY USED ON APPLICATION				D	30003
				SCALE: NONE	DRAWING NUMBER
					944AS110
					SHEET 1 OF 1

**DO NOT SCALE DRAWING**

**Figure 5. Typical Adhesive Used to Seal USRM Motor Tubes**

## 1. PLATE IN ACCORDANCE WITH TABLE III.



**Figure 6. PAD MK 74 Manifold Torque Test Item**

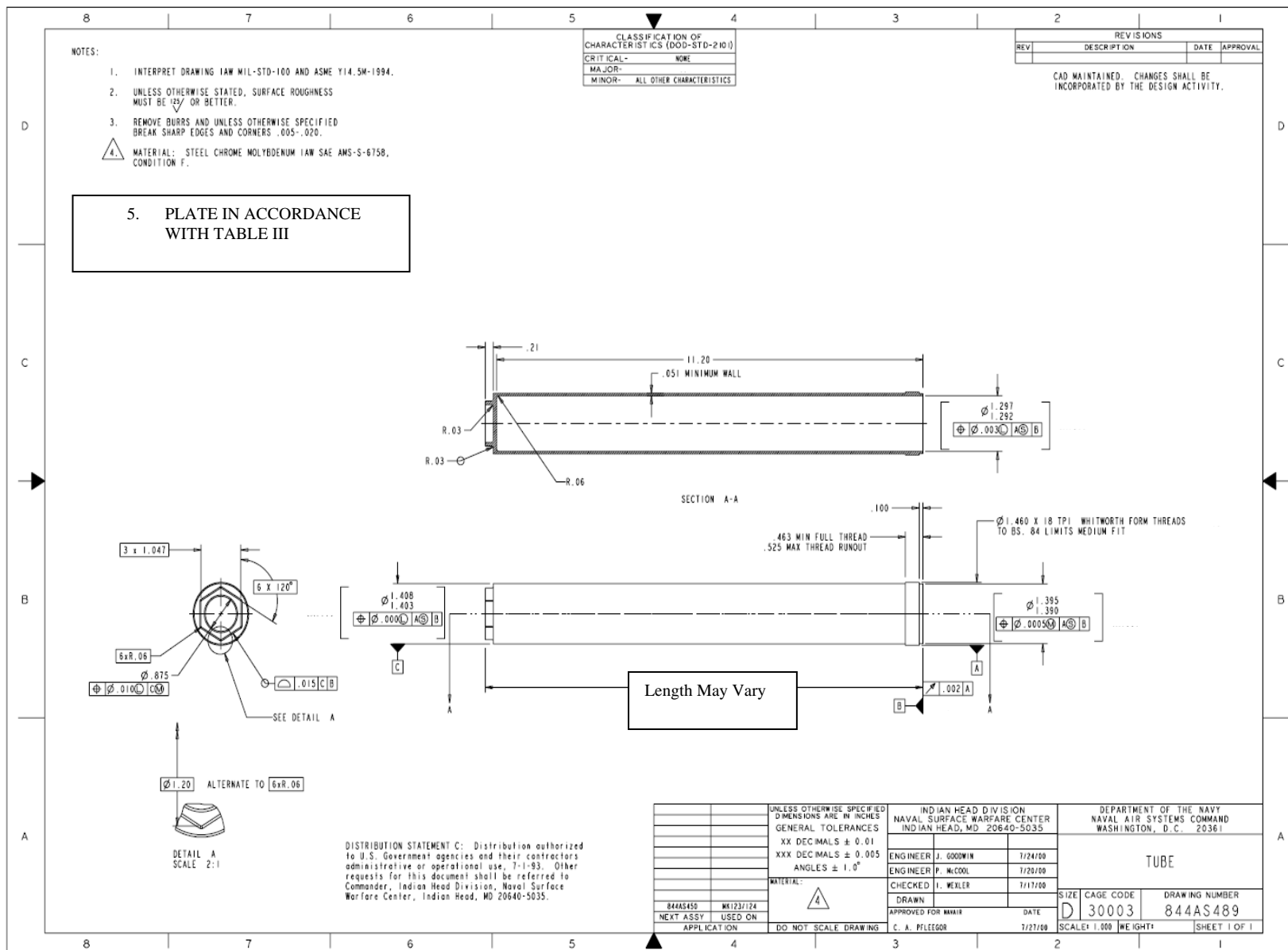
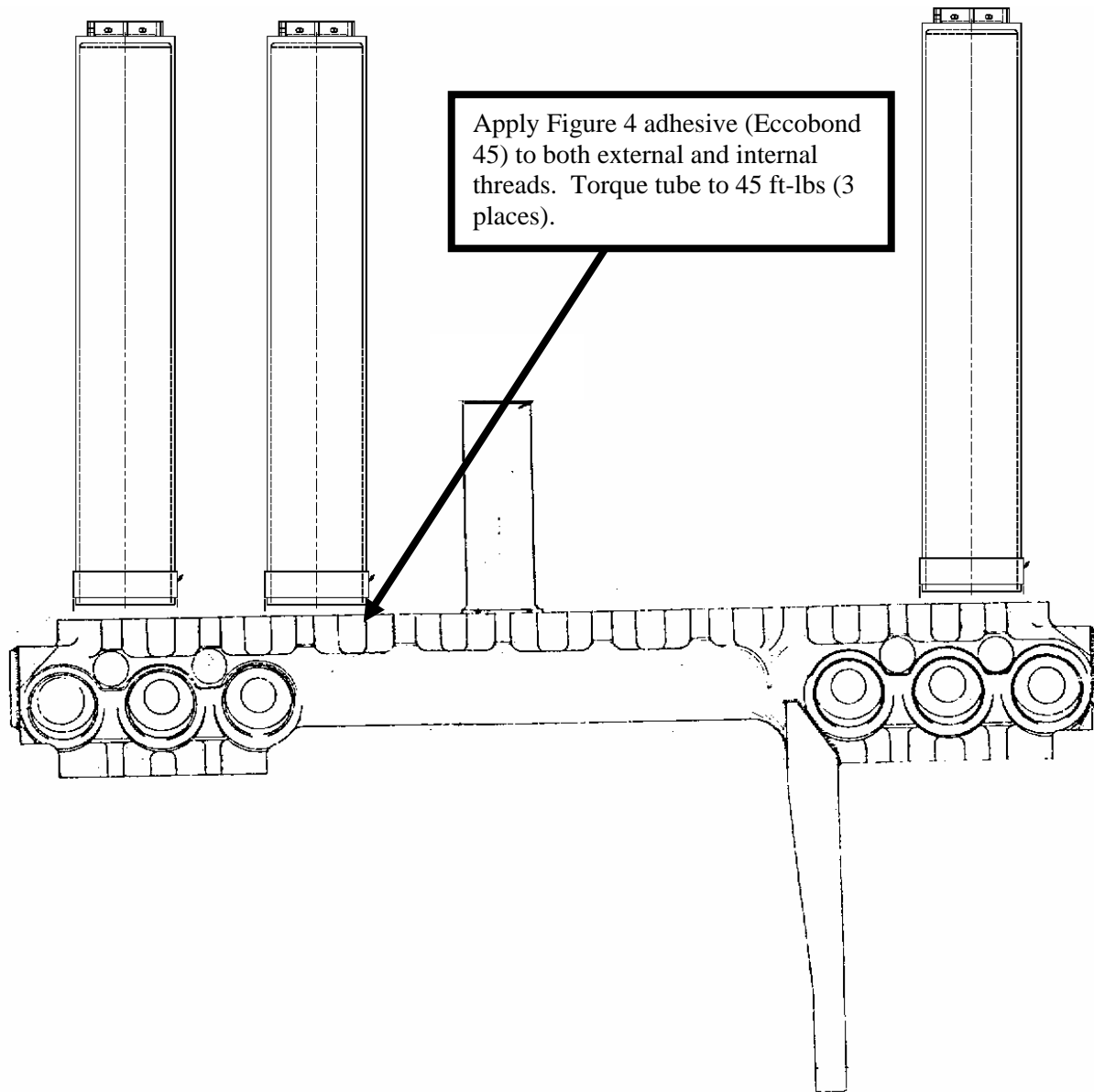


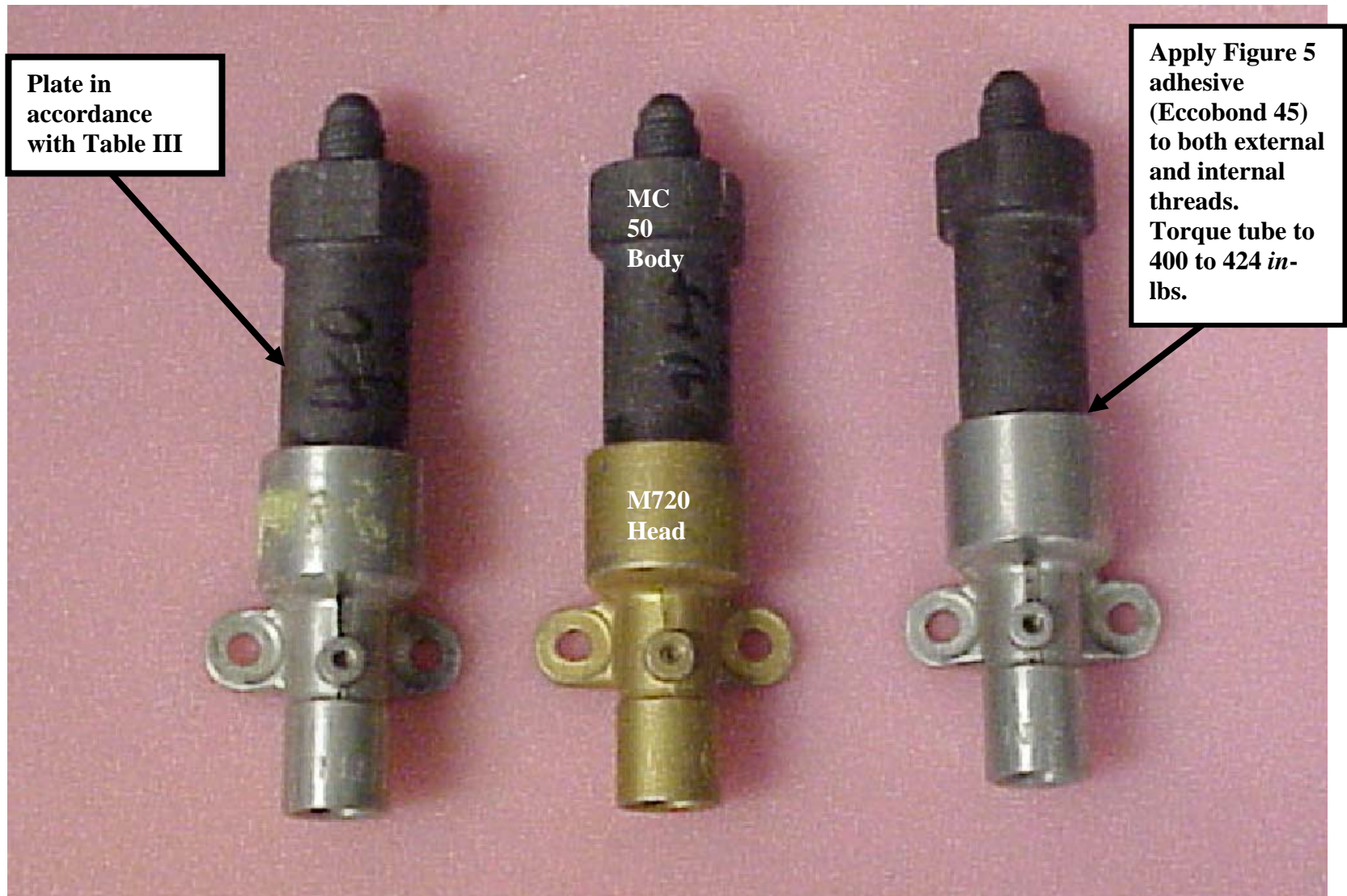
Figure 7. PAD MK 74 Manifold Tube Torque Test Item

**Table III. Plating Preparation for PADs and CADs**

Item designation	Quantity	Preparation description
Control	1 Mk 74 per Figure 4.	1. Strip and cadmium plated as per MIL-STD-870[24], Type II, Class 1, with plating .0005 in (0.013 mm) to .0007 (0.018 mm) thick on all surfaces. 2. Heat treat, to embrittlement relieve, as soon as possible after plating, but no later than 4 hours after plating at 374°F to 446°F (190°C to 230°C) for at least 24 hours.
	3 Motor tubes per Figure 5.	
	3 MC 50 (see Figure 6)	
(12) Trivalent coating Low-nickel	1 Mk 74 per Figure 4.	Strip and plate panel in accordance with drawing 513-174-0213 (Figure 6).
	3 Motor Tubes per Figure 5	
	3 MC 50 (see Figure 6)	
(12x) Trivalent coating High-nickel	1 Mk 74 per Figure 4. 3 Motor tubes per Figure 5.	Strip and plate panel in accordance with drawing SK07005-E213K-1 (Figure 7).



**Figure 8. Modified MK 74 to Motor Tube Test Assembly**



**Figure 9. CAD Torque Test Assembly**



CLASSIFICATION OF CHARACTERISTICS (DOD-STD-210)				REVISIONS			
CRITICAL - NONE				REV	DESCRIPTION	DATE	APPROVAL
MAJOR: 4							
MINOR: ALL OTHER CHARACTERISTICS							

CAD MAINTAINED, CHANGES SHALL BE INCORPORATED BY THE DESIGN ACTIVITY

NOTE:

1. INTERPRET DRAWING IN ACCORDANCE WITH ASME Y14.100-2004 WITH APPENDICES B, C, D, AND E.
- (M101) 2. STRIP ALL SURFACES TO BARE METAL AND CLEAN PER ASTM B 322-99. ALL CLEANING AND ANY STRIPPING PROCESS SHALL BE CYANIDE FREE.
- (M102) 3. BAKE IN ACCORDANCE WITH SAE AMS 2759/9A WITHIN THREE HOURS AFTER STRIPPING. AFTER STRESS RELIEF BAKE AND BEFORE ANY PLATING, ALL SURFACES SHALL REMAIN HYDROCARBON OIL FREE.
4. PROTECT FROM MOISTURE BEFORE DESCALING. DESCAL PER MIL-S-5002D AMENDMENT 2 AND THEN IMMEDIATELY CLEAN PER ASTM D 322-99. DESCALING AND CLEANING SHALL BE CYANIDE FREE.

NON-MANDATORY ADVISORY INFORMATION:  
A LOW TOXIC RUST REMOVER WAS FOUND AS AN EFFECTIVE CYANIDE FREE DESCALER IN SOME APPLICATIONS.  
RUST REMOVER GDP 1000  
GLOBAL DIVERSIFIED PRODUCTS  
11400 47TH ST.  
N. CLEARWATER FL 33767  
(727) 571 3777

- (M103) 5. IMMEDIATELY AFTER NOTE 4 CLEANING, ACTIVATE AND NICKEL-PHOSPHORUS COAT PER ASTM B 733-97, TYPE V, CLASS 1, SERVICE CONDITION SC0, TO 0.0001 THICKNESS MAXIMUM.
- (M104) 6. ZINC-NICKEL PLATE IN ACCORDANCE WITH ASTM B841-99, CLASS 1, WITH 5% TO 7% NICKEL. BAKE IN ACCORDANCE WITH SAE AMS 2759/9A WITHIN THREE HOURS AFTER PLATING. MAXIMUM BAKING TIME SHALL BE 26 HOURS.
7. ZINC PHOSPHATE CONVERSION COAT PER ASTM D2092-95 METHOD A.
8. TRIVALENT CHROMATE CONVERSION COAT PER MIL-DTL-81706B, TYPE II, CLASS 1A, FORM 1, METHOD C, (M817062A1C) USING DRAWING 513-174-0216 QUALIFIED SOURCE(S).

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GENERAL TOLERANCES					
XX DECIMALS ± —					
XXX DECIMALS ± —					
ANGLES ± —					
MATERIAL:					
NEXT ASSY USED ON APPLICATION					

ENGINEER	JLA 2r	2006-10-19
ENGINEER		
CHECKED	DMA	2006-10-19
DRAWN	TLG	2006-10-19
APPROVED FOR SHIP		DATE
		2006-10-20

SIZE	CAGE CODE	DRAWING NUMBER
D	14083	513-174-0213
SCALE: NONE	SHEET 1 OF 1	

**Figure 10. Zinc-Nickel Plating, Low-Nickel, Low Embrittlement, Hexavalent-Chromate Free Rework (ID 12)5,11,8,9,10,12,13**

CLASSIFICATION OF CHARACTERISTICS (DDD-STD-2100)				REVISIONS			
CRITICAL- NONE MAJOR- 4 MINOR- ALL OTHER CHARACTERISTICS				REV	DESCRIPTION	DATE	APPROVAL
CAD MAINTAINED. CHANGES SHALL BE INCORPORATED BY THE DESIGN ACTIVITY							
<p>NOTE:</p> <p>1. INTERPRET DRAWING IN ACCORDANCE WITH ASME Y14.100-2004 WITH APPENDICES D, C, D, AND E.</p> <p>(M101) 2. STRIP ALL SURFACES TO BARE METAL AND CLEAN PER ASTM B 322-99. ALL CLEANING AND ANY STRIPPING PROCESS SHALL BE CYANIDE FREE.</p> <p>(M102) 3. BARE IN ACCORDANCE WITH SAE AMS 2759/9A WITHIN THREE HOURS AFTER STRIPPING. AFTER STRESS RELIEF BARE AND BEFORE ANY PLATING, ALL SURFACES SHALL REMAIN HYDROCARBON OIL FREE.</p> <p>4. PROTECT FROM MOISTURE BEFORE DESCALING. DESCALE PER MIL-S-50020 AMENDMENT 2 AND THEN IMMEDIATELY CLEAN PER ASTM B 322-99. DESCALING AND CLEANING SHALL BE CYANIDE FREE.</p> <p>NON-MANDATORY ADVISORY INFORMATION: A LOW TOXIC RUST REMOVER WAS FOUND AS AN EFFECTIVE CYANIDE FREE DESCALER IN SOME APPLICATIONS. RUST REMOVER GDP-1000 GLOBAL DIVERSIFIED PRODUCTS 11400 47TH ST. N. CLEARWATER FL 33767 (727) 571 3777</p> <p>(M103) 5. IMMEDIATELY AFTER NOTE 4 CLEANING, ACTIVATE AND NICKEL-PHOSPHORUS COAT PER ASTM B 733-97, TYPE V, CLASS 1, SERVICE CONDITION SCO, TO 0.0001 THICKNESS MAXIMUM.</p> <p>(M104) 6. ZINC-NICKEL PLATE IN ACCORDANCE WITH SAE AMS 2417G, TYPE 3, WITH 9% TO 13% NICKEL (12% NOMINAL). BARE IN ACCORDANCE WITH SAE AMS 2759/9A WITHIN THREE HOURS AFTER PLATING. MAXIMUM BAKING TIME SHALL BE 26 HOURS.</p> <p>7. ZINC PHOSPHATE CONVERSION COAT PER ASTM D2092-95 METHOD A.</p> <p>8. TRIVALENT CHROMATE CONVERSION COAT PER MIL-DTL-81706B, TYPE 1, CLASS 1A, FORM 1, METHOD C, (M817062A1C) USING DRAWING 513-174-0216 QUALIFIED SOURCE(S).</p>							
<p>DISTRIBUTION STATEMENT C: Distribution authorized to U.S. Government agencies and their contractors administrative or operational use, 7-1-93. Other requests for this document shall be referred to Commander, Indian Head Division, Naval Surface Warfare Center, Indian Head, MD 20640-5035.</p>							
<p>UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES GENERAL TOLERANCES XX DECIMALS ± — XXX DECIMALS ± — ANGLES ± —</p> <p>MATERIAL:</p> <p>NEXT ASSY USED ON APPLICATION DO NOT SCALE DRAWING</p>				<p>INDIAN HEAD DIVISION NAVAL SURFACE WARFARE CENTER INDIAN HEAD, MD 20640-5035</p> <p>ENGINEER <i>John A. Whelan</i> 1-23-2007 ENGINEER <i>Ar. Dore</i> 1/23/07 CHECKED</p> <p>DRAWN <i>TC</i> 1/23/07 APPROVED FOR NAVAIR <i>GP. Chish</i> 1/24/07</p>			
				<p>DEPARTMENT OF THE NAVY NAVAL SURFACE WARFARE CENTER INDIAN HEAD, MD 20640-5035</p> <p>ZN-NI PLATING, HIGH NICKEL, LOW EMBRITTLEMENT, HEXAVALENT CHROMATE FREE, REWORK</p> <p>SIZE CAGE CODE DRAWING NUMBER D 14083 SK07005-E213K-1</p> <p>SCALE: NONE SHEET 1 OF 1</p>			

Figure 11. Zinc-Nickel Plating, High-Nickel, Low Embrittlement, Hexavalent-Chromate Free Rework (ID 12x)5,11,8,9,14,12,13

## TEST RESULTS AND DISCUSSION

### Environmental Testing

#### Test Panel Rain, Humidity, Cyclic Salt Fog

Figures 12 and 13 show results from the six TCP panels designated 12-1, 12-2, 12-3, 12x-1, 12x-2, and 12x-3 subject to rain, humidity and 1000 hours of cyclic salt-fog testing per Table II, displayed no apparent damage from chalking or rusting. Figure 14 shows 2000 and 3000 hours cyclic salt fog testing. Although these panels were not evaluated for blistering, checking, cracking, flaking, or filiform corrosion, these panels displayed no apparent damage of any type. By comparison, the untreated control panels exhibited increasing levels of rust. It became totally covered by thick corrosion by the 1000-hour salt-fog evaluation. After 3000 hours, the control lost 36% of its original weight to rust. White residue observed on the panels during salt-fog testing are artifacts of salt deposits related to the test environment and not damage to sample's surface. Whiter residue appears on the low-nickel panels more than the high-nickel panels.

These results are superior to cadmium with CCC or zinc high-nickel with CCC shown in IHTR 2694, Table XI<sup>3</sup>.

#### Test Panel Sulfur Dioxide and Cyclic Salt Fog Unpainted

Figure 15 shows the progress of corrosion during sulfur dioxide/salt fog testing. Figure 16 shows that the higher nickel content fairs somewhat better at 4 days than the low nickel content. Figure 15 and 17 show that all panels exhibited substantial red rust in one week.

#### Test Panel Sulfur Dioxide and Cyclic Salt Fog Painted

Figure 18 illustrates a comparison between painted cadmium plated panels and painted TCP on low zinc nickel. These painted CCC on cadmium plated panel resist blistering and score corrosion the best. Painted TCP on low zinc nickel that was previously rain/humidity/salt fog tested exhibited only moderate blistering and score corrosion throughout the 78 day testing.

Figure 19 illustrates how effective TCP is in reducing corrosion in sulfur dioxide testing. The second panel from the left has substantially more corrosion than the two TCP panels on the right. Also note that the cadmium panel has no blistering whereas the three panels on the right have blisters.

#### Test Panel Chip Resistance

Table IV shows evaluations on the five TCP panels (three as the control with TCP over zinc-nickel (5 percent), and two with TCP over zinc-nickel (12 percent) subjected to chip resistance testing, indicated







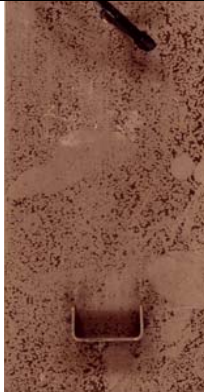
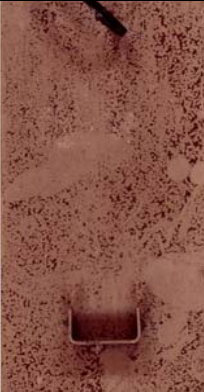
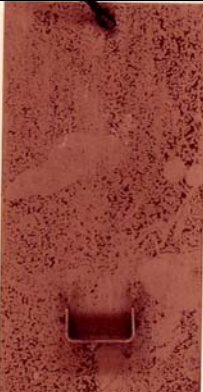





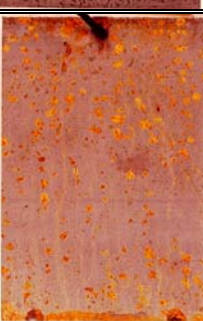
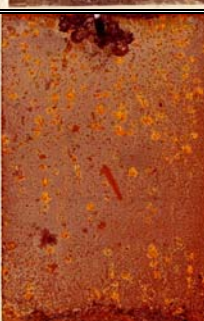


minimal and acceptable damage to panels with both TCP treatments.) Both groups of panels displayed similar chipping resistance ratings for both treatments. Therefore, the 12 percent nickel TCP receives a “pass” for this test.

### **Torque Retention**

Table V shows results from the tube breakaway torque tests. All the assemblies required more torque to unscrew than the torque used to assemble them.

### **Compatibility**

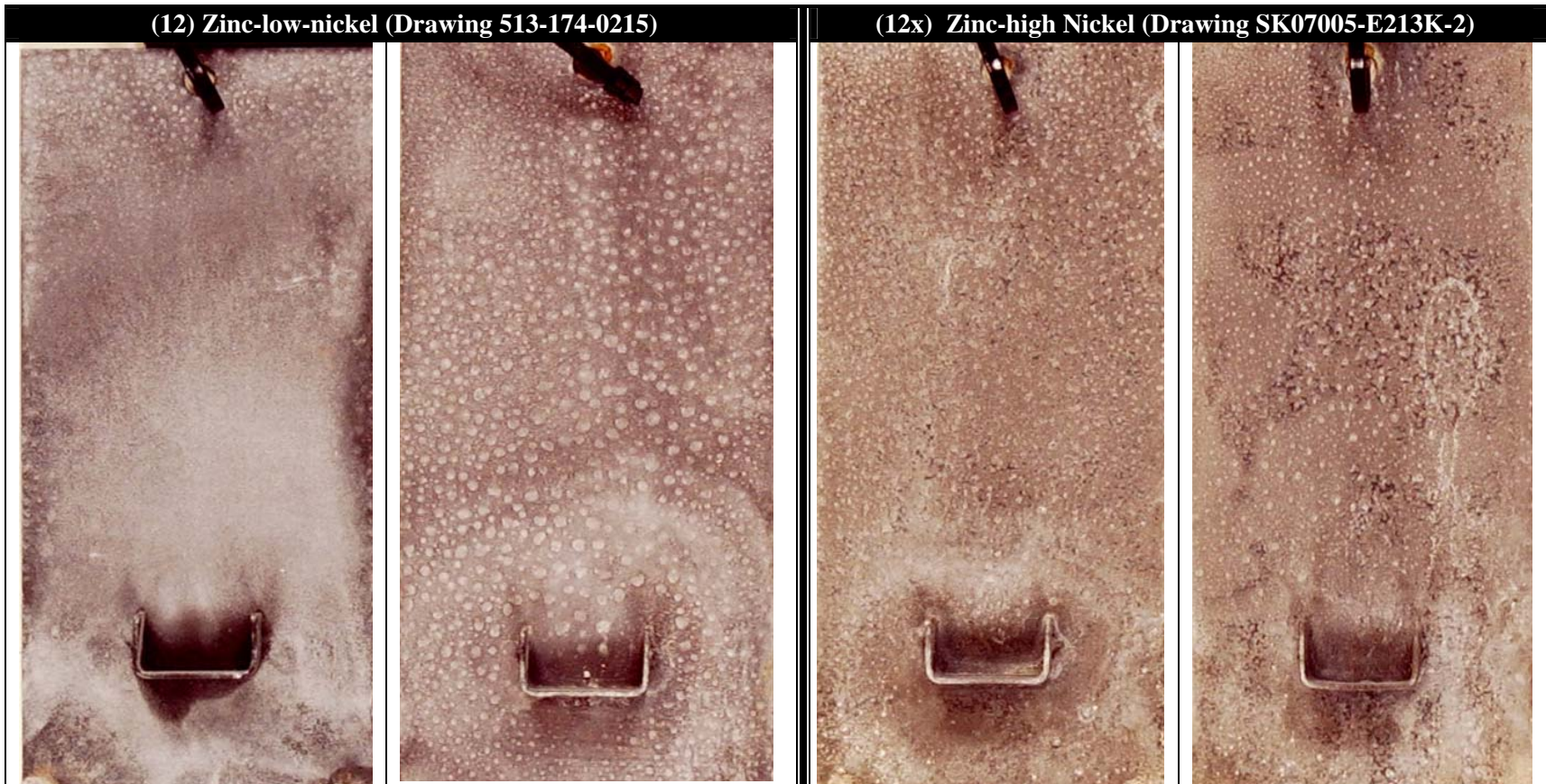
Appendix A contains a series of differential scanning calorimetry (DSC), and Thermogravimetric Analysis compatibility test. They revealed that TCP is compatible with Mechanite 19. Mechanite 19 is a double-base (nitrocellulose/nitroglycerine) propellant used in some CAD/PAD items.

	As Received	Rain Test	10 Day Humidity	168 Hr	1000 Hr	1000 Hr Rear View
Panel 12-2 (TCP/zinc-low-nickel)						
Panel 12x-1 (TCP/zinc-high-nickel)						
Control (uncoated)						

Note: All ASTM G85 salt fog tests were performed in Auto-Technology Model CCT-NC-20.

















**Figure 12. Photos of TCP/Zinc-Nickel/Steel Panels After Various Stages of Salt Fog Testing**





Note: All ASTM G85 salt fog tests were performed in Auto-Technology Model CCT-NC-20.















**Figure 13. 1000 Hr Cyclic Salt Fog Test of TCP/Zinc-Nickel/Steel Panels**

	2000 Hrs			3000 Hrs		
<b>Panel 12-2 (TCP/Zinc- low-nickel)</b>						
<b>Panel 12x-1 (TCP/Zinc- High Nickel)</b>						
<b>Control (Uncoated)</b>						<b>36% weight loss after glass bead blasting 3000hr control</b>

Note: All sulfur dioxide salt fog tests were performed in a Harshaw Environmental Test Chamber of Engehard Corporation, Cleveland, OH.

**Figure 14. 2000 and 3000 Hr Cyclic Salt Fog Test of TCP/Zinc-Nickel/Steel Panels**

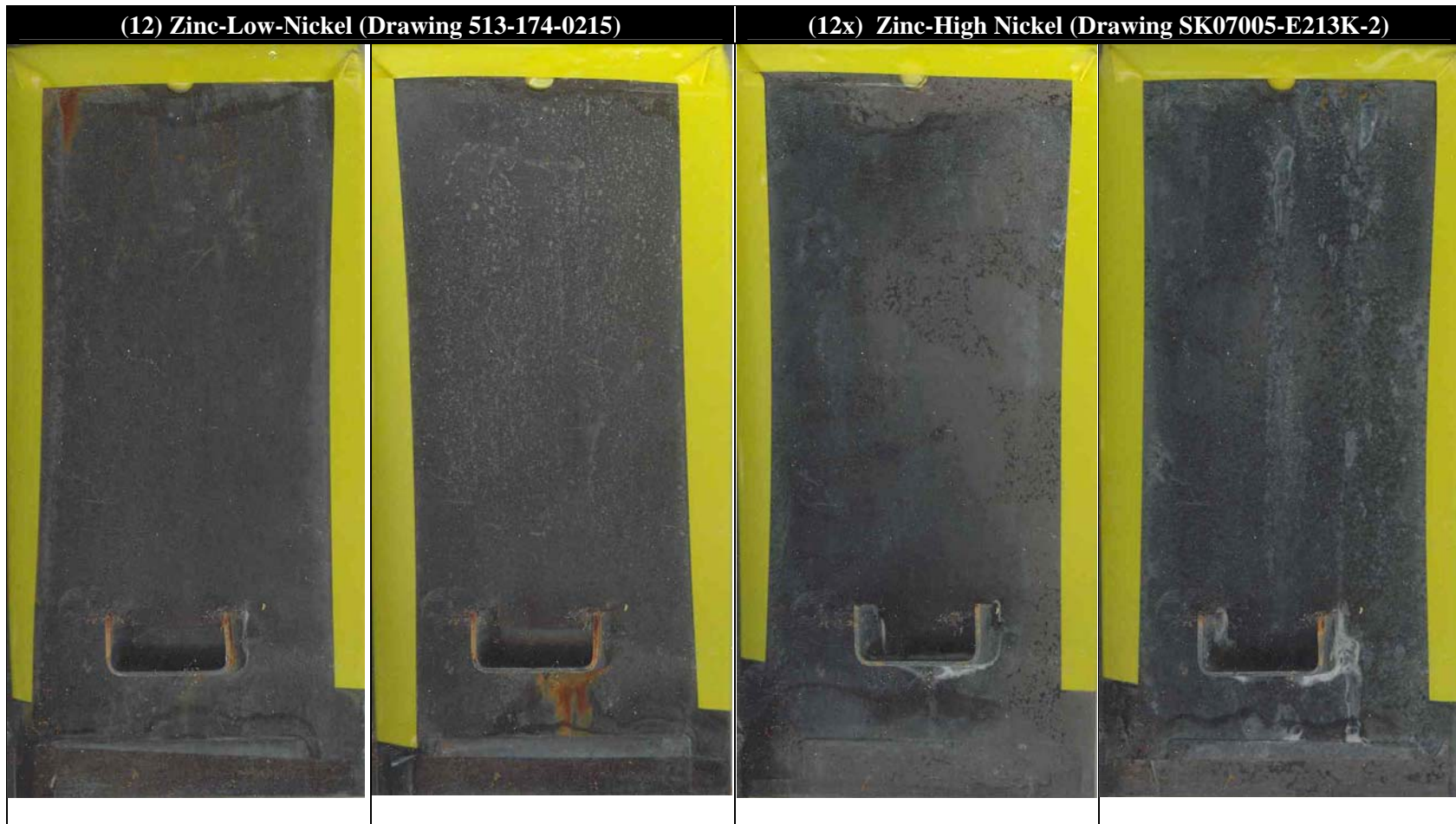


	As Received	24 Hr	48 Hr	72 Hr	96 Hr	168 Hr
<b>Panel (12) (TCP/zinc- low-nickel)</b>						
<b>Panel (12x) (TCP/zinc- high-nickel)</b>						
<b>Control (uncoated)</b>						

Note: All sulfur dioxide salt fog tests were performed in a Harshaw Environmental Test Chamber of Engehard Corporation, Cleveland, OH.

**Figure 15. Sulfur Dioxide/Salt Fog Testing on TCP/Zinc-Nickel/Steel Panels After Various Stages**





Note: All sulfur dioxide salt fog tests were performed in a Harshaw Environmental Test Chamber of Engehard Corporation, Cleveland, OH.





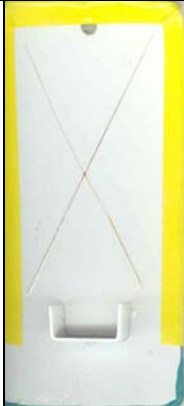











**Figure 16. 92 Hr (4 days) Sulfur Dioxide/Salt Fog Testing on TCP/Zinc Nickel**



Note: All sulfur dioxide salt fog tests were performed in a Harshaw Environmental Test Chamber of Engehard Corporation, Cleveland, OH.




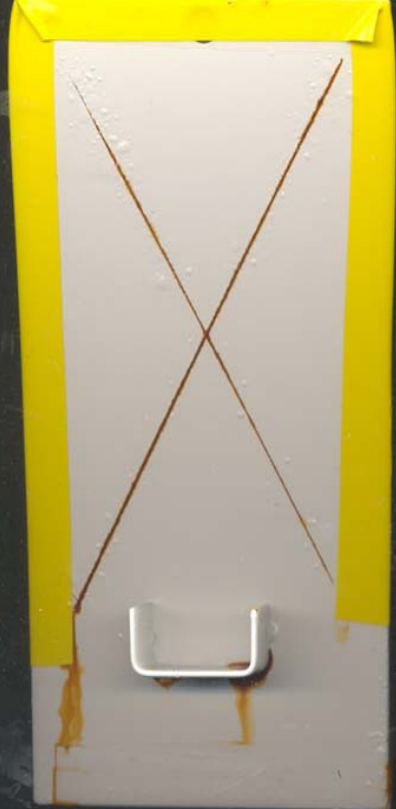
**Figure 17. 168 Hr (1 Week) Sulfur Dioxide/Salt Fog Testing on TCP/Zinc Nickel**



	As Received	168 Hr	336 Hr	504 Hr	672 Hr	840 Hr	1008 Hr	1872 Hr
Painted Cadmium (Control)								
Cadmium plate as per MIL-STD-870[24], Type II, Class 1, with plating .0005 (0.013 mm) to .0007 (0.018 mm) thick on all surfaces. Then heat treat, to embrittlement relieve, as soon as possible after plating, but no later than 4 hr after plating at 374 °F to 446 °F (190 °C to 230 °C) for at least 24 hr. Prime and Paint as per MIL-DTL-85097[15]. Primer coating shall be per MIL-P-53030[16]. Paint shall be as per MIL-PRF-85285[17], Color, Gloss White No. 17935 per FED-STD-595[18].								
Previously subject to rain test, 10 days humidity and 120 days salt fog then freshly scored before Sulfur dioxide/salt fog testing (used 6p23 in <a href="#">IHTR 2694 Table VIII</a> )								
Plated per drawing 513-174-0215. Prime and Painted per MIL-DTL-85097[15]. Primer coating per MIL-P-53030[16]. Paint per MIL-PRF-85285[17], Color, Gloss White No. 17935 per FED-STD-595[18].								


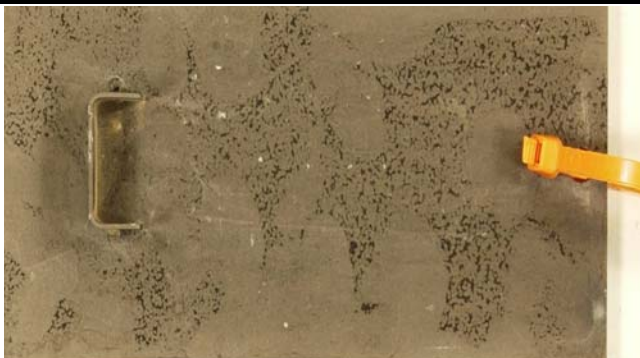
Note: All sulfur dioxide salt fog tests were performed in a Harshaw Environmental Test Chamber of Engehard Corporation, Cleveland, OH.

**Figure 18. Painted After Various Stages Of Sulfur Dioxide/Salt Fog Testing Per Table II**

Cadmium Plated per Table III (Control)	No TCP (Step 8) on Zinc-Low-Nickel (Drawing 513-174-0215)	TCP on Zinc-Low-Nickel (Drawing 513-174-0215)	
			
<p>Prime and painted per MIL-DTL-85097[15]. Primer coating per MIL-P-53030[16]. Paint per MIL-PRF-85285[17], Color, Gloss White No. 17935 per FED-STD-595[18].</p> <p>Note: All sulfur dioxide salt fog tests were performed in a Harshaw Environmental Test Chamber of Engehard Corporation, Cleveland, OH.</p>			






**Figure 19. Photos of Painted after 1872 Hrs (78 days) Sulfur Dioxide/Salt Fog Testing**

**Table IV. Chip Resistance Test TCP/Zinc-Nickel Results**

Designation	Typical panels  Coating (treatment) chipping resistance test procedure: ASTM D3170-03[22]  Testing performed by: Graig Jolley, NSWCCD Code 614 Test date: 17 August 2007  Panels evaluation performed by: Dan Davis, NSWCCD Code 613	Panel ID Chip No. Rating <sup>1</sup>	Chip Size Rating <sup>1</sup>	Pass/Fail
12-4		7	A	Pass
12-5		6	A	Pass
12-6		6	A	Pass
(Plated per drawing 513-174-0215 (Figure 1))				
12x-4		6	A	Pass
12x-5		6	A	Pass
(Plated per drawing SK07005-E213K-1 (Figure 2))				

<sup>1</sup>Chip number and size ratings evaluated per ASTM D3170-03[23] and SAE J400[25] (Nov. 2002 Rev), using the "physical count method" (number of chips of each size).

**Table V. Unscrew Torque Typical PADs and CADs Test Results<sup>1</sup>**

Designation	PADs: Underseat rocket motor manifold/tubes (See Figure 8)	Unscrew torque ft-lb	As a percent of 45 ft-lb applied torque <sup>2</sup>
<b>(12)</b> <b>Zinc-low nickel plated per 513-174-0213 (Figure 10)</b>		90	200%
		120	270%
		120	270%
		Avg: 110 $\sigma$ 17	240%
<b>(12x)</b> <b>Zinc-high nickel plated per SK07005-E213K-1 (figure 11)</b>		95	210%
		85	190%
		100	220%
		Avg: 93 $\sigma$ 8	210%
<b>Figure 8; Cadmium plate per table III (control)</b>		80	180%
		110	240%
		125	280%
		Avg: 105 $\sigma$ 23	230%
Designation	CADs: MC 50 Steel Body (See Figure 9)	Unscrew torque in-lb	As a percent of Avg. 400-425 in-lb Applied Torque <sup>2</sup>
<b>(12)</b> <b>Zinc-low nickel plated per 513-174-0213 (Figure 10)</b>		960	230%
		480	120%
		900	220%
		Avg: 780 $\sigma$ 260	190%
<b>(Control)</b> <b>cadmium plate per table III</b>		1500	360%
		840	200%
		1560	380%
		Avg: 1300 $\sigma$ 400	320%

<sup>1</sup> All assemblies cured one week at room temperature after assembly.<sup>2</sup> Torque wrench serial number is 11389. Calibration due 06-16-2007.

## CONCLUSION

- Zinc-Nickel plate on 4130 steel prepared in accordance with 513-174-0215 or 513-174-0213 is qualified for CAD/PAD service provided it is compatible with the materials it may be in contact in the CAD/PAD.
- TCP/zinc-phosphate conversion coating offers a low toxicity alternative to hexavalent chromate on zinc-nickel plated 4130 steel. The TCP without zinc-phosphate on zinc-nickel plated steel is unlikely to provide corrosion protection due to the porous nature of zinc-nickel and the aqueous nature of TCP corroding from within these pores. This is based on testing by the TCP's co-inventor and previous testing using aqueous Teflon® to replace hexavalent chromate (See IHTR 2694, Table XII)[3].
- TCP is compatible with Mechanite 19 propellant.
- TCP/zinc-phosphate/zinc-low-nickel is qualified for all PADs and CADs assuming it is compatible with the various materials (i.e., propellants) used in the same.
- TCP/zinc-phosphate/zinc-high nickel has somewhat better corrosion resistance in sulfur dioxide/salt fog environments than TCP/zinc-phosphate/zinc-low-nickel.
- TCP/zinc-phosphate/zinc-low-nickel offers much better corrosion protection than CCC/cadmium or CCC/zinc-low-nickel.
- TCP/zinc-phosphate/zinc-nickel is non-proprietary.
- TCP/zinc-phosphate/zinc-low/high nickel plating on thread wetted with Eccobond 45 adhesive is unlikely to lose preload torque after cure.
- TCP/zinc-phosphate/zinc-low or high-nickel plated CAD can endure shipboard marine application provided exposure to sulfur dioxide is not too extreme.
- TCP/zinc-phosphate/zinc-low or high-nickel plated CAD can endure long term exterior marine applications even unpainted (painted TCP/zinc-phosphate/zinc-high nickel was *never* tested).
- TCP/Zn-P/Zn-Ni used as prescribed in Figures 1, 2, 10, and 11 exceeds all of our objectives.
- TCP/Zn-P/Zn-Ni used as prescribed in Figures 1, 2, 10, and 11 is qualified to replace CCC/Cd on steel CADs and PADs, provided it is compatible with materials it would be exposed to in the CAD or PAD (e.g., propellant, energetics, dissimilar material etc.).

## RECOMMENDATIONS

- If the trivalent chromate described in this report is compatible with materials it would be exposed to in a CAD or PAD, replace the hexavalent chromate/cadmium plating on steel PAD or CAD surfaces with trivalent chromate/zinc phosphate/zinc nickel/nickel strike in accordance with drawing number:
  - 513-174-0215 (Figure 1) on new parts
  - 513-174-0213 (Figure 10) on reworked parts
  - SK07005-E213K-2 (Figure 2) on new parts subject to severe SO<sub>2</sub> fog on unpainted surfaces
  - SK07005-E213K-1 (Figure 11) on reworked parts subject to severe SO<sub>2</sub> fog on unpainted surfaces.
- This plating is thicker than cadmium plating. Therefore, if final part dimensions apply after plating, assure that this coating system does not violate any minimum wall or size requirements before any final production.
- Replace cadmium plating on steel in all CAD/PAD rework specifications as recommended in the first and second bullets.
- Replace cadmium plating on steel in all production CAD/PAD technical data packages as recommended in the first and second bullets.



## REFERENCES

1. US Department of Labor Occupational Safety and Health Administration, Hexavalent Chromium Standards, Section 5(a)(1) and 5(a)(2) of the OSHA Act, February 28, 2006.
2. Federal Register / Vol. 71, No. 209 / Monday, October 30, 2006, Rules and Regulations, Page 63238, 29 CFR 1910.
3. *Cadmium Replacement For Propellant Actuated Devices (PADS)*, IHTR 2694, July 15, 2005.
4. *Protective Coating Analysis*, Sabal, Diane L. and Dohm, Gregory J., SAFE Journal 35, p209-p217, 1997.
5. ASME Y14.100-2000, *Engineering Drawing Practices*, November 23, 2001.
6. ASTM B 322, *Standard Guide for Cleaning Metals Prior to Electroplating*, November 10, 1999.
7. ASTM B 849-94, *Standard Specification for Pre-Treatments of Iron or Steel for Reducing Risk of Hydrogen Embrittlement*, July 15, 1994.
8. MIL-S-5002, *Surface Treatments and Inorganic Coatings for Metal Surfaces of Weapons Systems*, October 20, 1999.
9. ASTM B 733 - 97 *Standard Specification for Autocatalytic (Electroless) Nickel-Phosphorus Coatings on Metal*, July 10, 1997.
10. ASTM B 849-99, *Standard Specification for Pre-Treatments of Iron or Steel for Reducing Risk of Hydrogen Embrittlement*, November 10, 1999.
11. SAE AMS 2759/9A, *Hydrogen Embrittlement Relief (Baking) of Steel Parts*, April 1, 2001.
12. ASTM D 2092, *Standard Guide for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting*, April 15, 1995.
13. MIL-DTL-81706B, *Chemical Conversion Materials for Coating Aluminum and Aluminum Alloys*, October 25, 2004.
14. SAE AMS 2417G, *Plating, Zinc-Nickel Alloy*, July 2004.
15. MIL-DTL-85097, *USN/USMC Propellant Actuated Devices (PADS), General Specification for*, July 7, 2000.
16. MIL-P-53030, *Primer Coating, Epoxy, Water Reducible, Lead and Chromate Free*, March 9, 1992.
17. MIL-PRF-85285, *Coating: Polyurethane, Aircraft and Support Equipment*, April 30, 1997.

18. FED-STD-595, *Colors Used in Government Procurement*, December 15, 1989.
19. ASTM D 2247-99, *Standard Practice for Testing Water Resistance of Coatings in 100% Relative Humidity*, December 10, 1999.
20. MIL-P-83126A, *Propulsion Systems, Aircrew Escape, Design Specification for*, February 8, 1980.
21. ASTM G 85, *Standard Practice for Modified Salt Spray (Fog) Testing*, October 10, 2002.
22. ASTM D 3170, *Standard Test Method for Chipping Resistance of Coatings*, July 10, 2003.
23. SAE AMS-S-6758, *Steel, Chrome-Molybdenum (4130) Bars and Reforging Stock (Aircraft Quality)*, July 1, 1998.
24. MIL-STD-870, *Cadmium Plating, Low Embrittlement, Electrodeposition*, July 30, 1986.
25. SAE J 403, *Chemical Compositions of SAE Carbon Steels-Supersedes Ford ESN-M1A46-A*, November 1, 2001.

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## **Appendix A**

### **MECHANITE 19 AND TCP COMPATIBILITY**

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8000  
Ser R312/008/dns  
19 Oct 2005

# MEMORANDUM

From: R312DS (D. Sorensen)  
To: E213K (H. Archer)  
Via: R312

Subj.: DSC/TGA THERMAL COMPATIBILITY TESTING OF MECHANITE 19 WITH CONVERSION COATINGS

Ref: (a) Lab Reference 200039569, Mechanite 19  
(b) Lab Reference 200039570, Cr (III) based conversion coating  
(c) Lab Reference 200039571, Cr (VI) based conversion coating  
(d) NATO STANAG #4147  
(e) CPIA Publ. 597, (1993) 301. "Compatibility of Energetic Materials by DSC and TG"

Enc: Nineteen thermal curves

## 1. Summary

Per your request, Differential scanning calorimetry (DSC), and Thermogravimetric Analysis (TGA) thermal compatibility testing was performed on the admixtures of Mechanite 19, ref (a), with two conversion coatings, ref (b) and (c). By the methods used, both conversion coatings are considered compatible via the methods used.

## 2. General Information on Compatibility

Under guidelines developed in ref (d) and (e), the definition of compatibility here is in terms of the relative predictive shelf life of the energetic component in intimate contact with another material. Thermal compatibility as determined by DSC or TGA involves running components individually and as admixtures. In DSC curves, the major decomposition peak is measured for the individual sample components and is compared with the major decomposition peak of the admixture. The greater the shift to lower temperatures for the admixture is the greater the degree of incompatibility between the ingredients. Reference (e) further develops the temperature shift guidelines per Table 1 to assess the degree of incompatibility in an admixture. It does not address changes to the Thermomechanical properties of the inert material, such as adhesive properties.

Degree of Incompatibility	DSC Peak Temperature Shift (°C)	TGA Change in Weight % (%mixtures- %ingred)
None	0-4	0-4
Slight	5-9	5-9
Small	10-19	10-19
Moderate	20-29	20-29
Large	≥ 30	≥ 30

## 3. DSC Analyses

All DSC analyses were performed on the TA Instruments Model 2910 after calibration against indium and zinc standards. The samples were run with a 50ml/min nitrogen purge at heating rates of 5°C/minute. Hermetically sealed aluminum pans were used in these tests. Hermetic pans are used since most

8000  
 Ser R312/008/dns  
 19 Oct 2005

weapon systems are sealed, simulating the worst-case scenario. Only single runs were used. In both cases, there was a  $<4^{\circ}\text{C}$  change upon adding the conversion coating to the energetic, so both conversion coatings are considered compatible via DSC.

Summary Table of DSC Compatibility Data at Heating Rate of  $5^{\circ}\text{C}/\text{minute}$ .

Sample	Exotherm Peak Temperature	Compatible
Mechanite 19	197.6°	
Cr (III) coating	No apparent rxn	
Cr (VI) coating	No apparent rxn	-
Mechanite 19 + Cr (III) coating	195.6°	$<4^{\circ}$ , Yes
Mechanite 19 + Cr (VI) coating	197.9°	$<4^{\circ}$ , Yes.

#### 4. TGA Analyses

Since two methods of determining compatibility are recommended per reference (d), TGA was also performed. All analyses were performed on the TA Instruments Model 2950. The samples were run with a 50ml/min nitrogen purge at heating rates of  $5^{\circ}\text{C}/\text{minute}$ . Pin-holed aluminum pans were used in these tests. In both cases, the first weight loss peaks circa  $193^{\circ}$ . The expected contributions from the isolated materials are larger than for the Cr(VI) admixture, so the admixture is compatible. For the Cr(III) admixture, the difference is negligible, so this admixture is deemed compatible as well.

Summary Table of TGA Compatibility Data at Heating Rate of  $5^{\circ}\text{C}/\text{minute}$ , 25:75 Ratios.

Sample	Temperature Region	% Wt loss	Compatible
Mechanite 19	50-192.34°, 50-195.33°	-45.47%, -50.26%	
Cr (III) coating	50-195.33°	0.45%	-
Cr (VI) coating	50-192.34°	1.01%	-
Mechanite 19 + Cr (III) coating	50-195.33°	-21.99%	Cf. -25.17%, Yes
Mechanite 19 + Cr (VI) coating	50-192.34°	-22.7%	Cf. -22.4%, Yes

5. Debra L. Knott assisted with the data collection. Please address any questions to the undersigned at extension 4671.

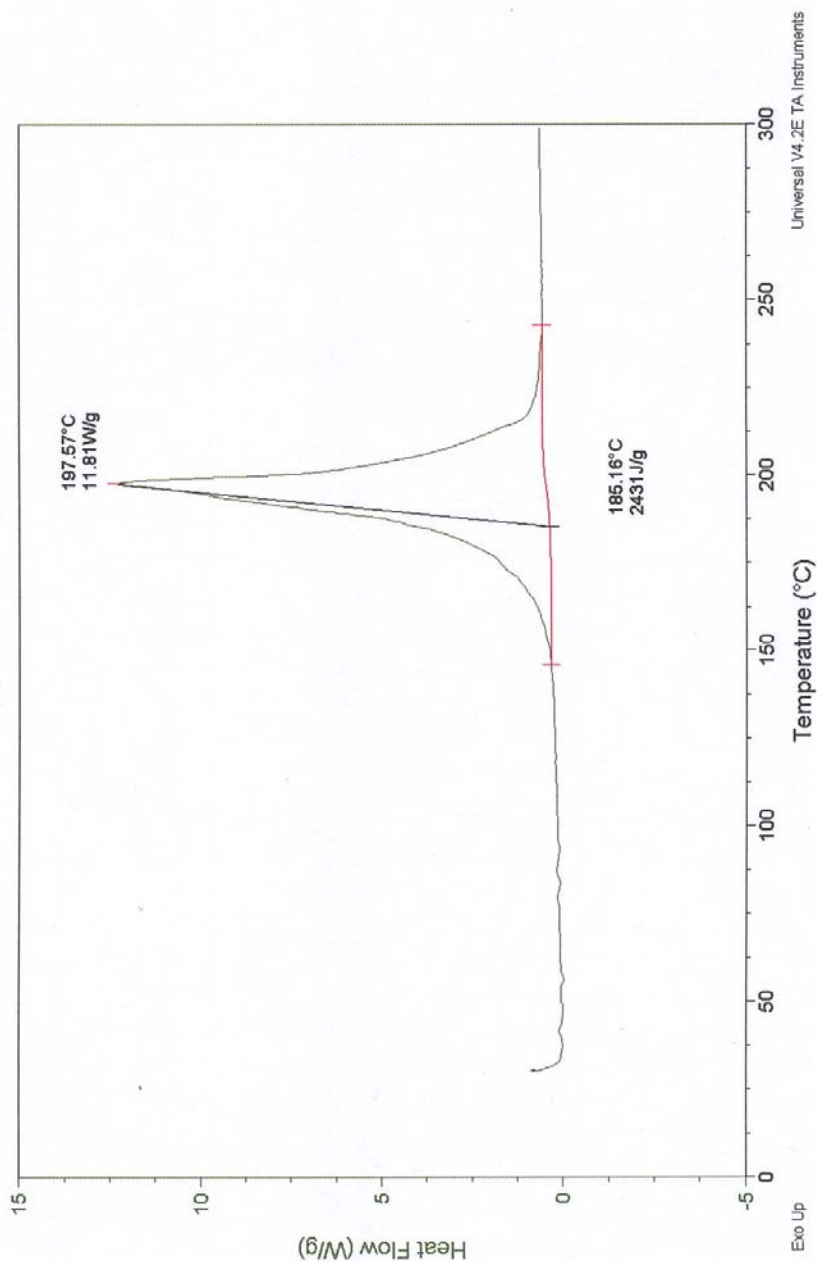
Daniel N. Sorensen, Ph.D.

8000  
Ser R312/008/dns  
19 Oct 2005

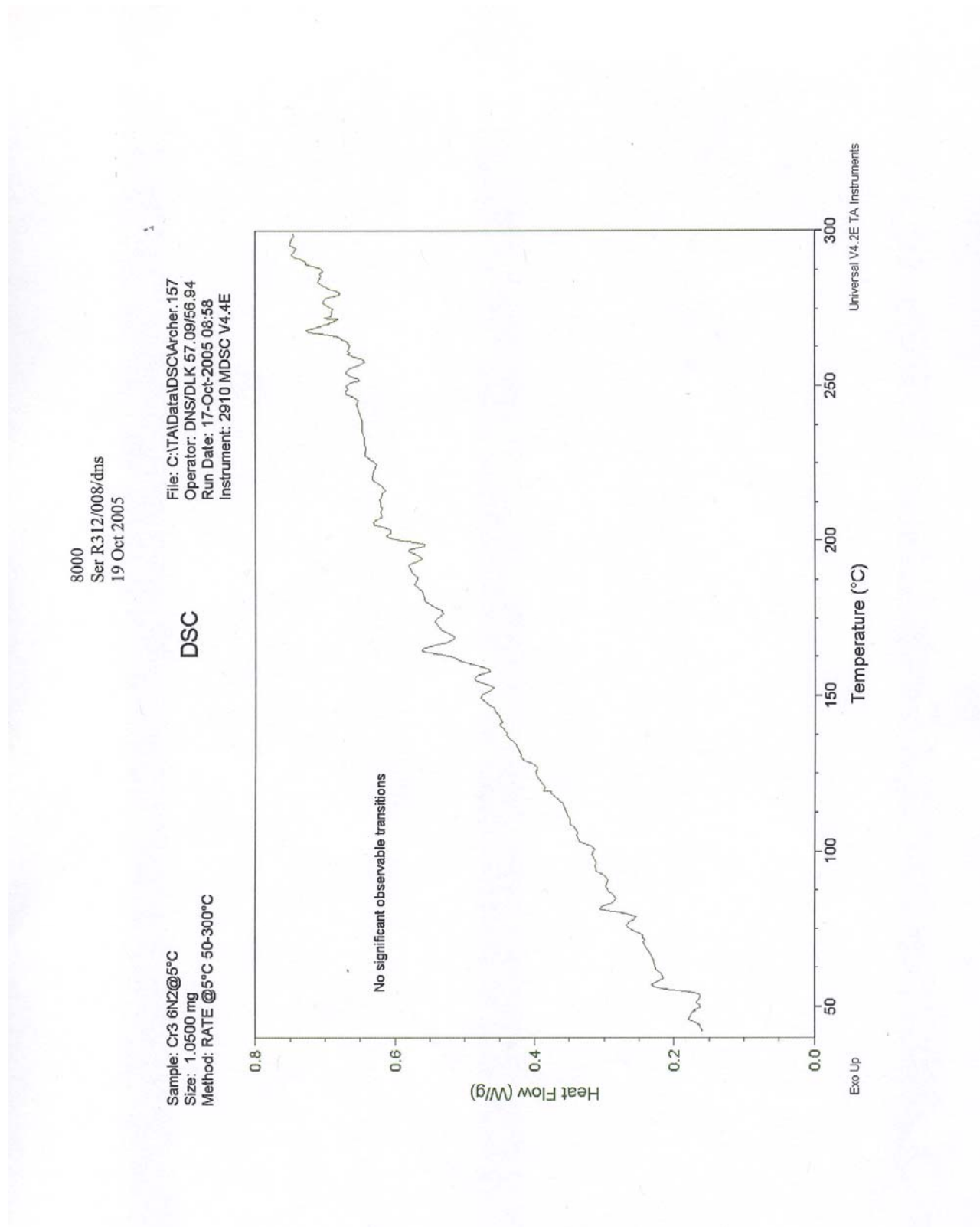
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Operator: DNS/DLK 57.05/56.94  
Run Date: 14-Oct-2005 15:13  
Instrument: 2910 MDSC V4.4E

# DSC

Sample: Mechanite 19@5°C  
Size: 1.0700 mg  
Method: RATE @5°C 50-300°C





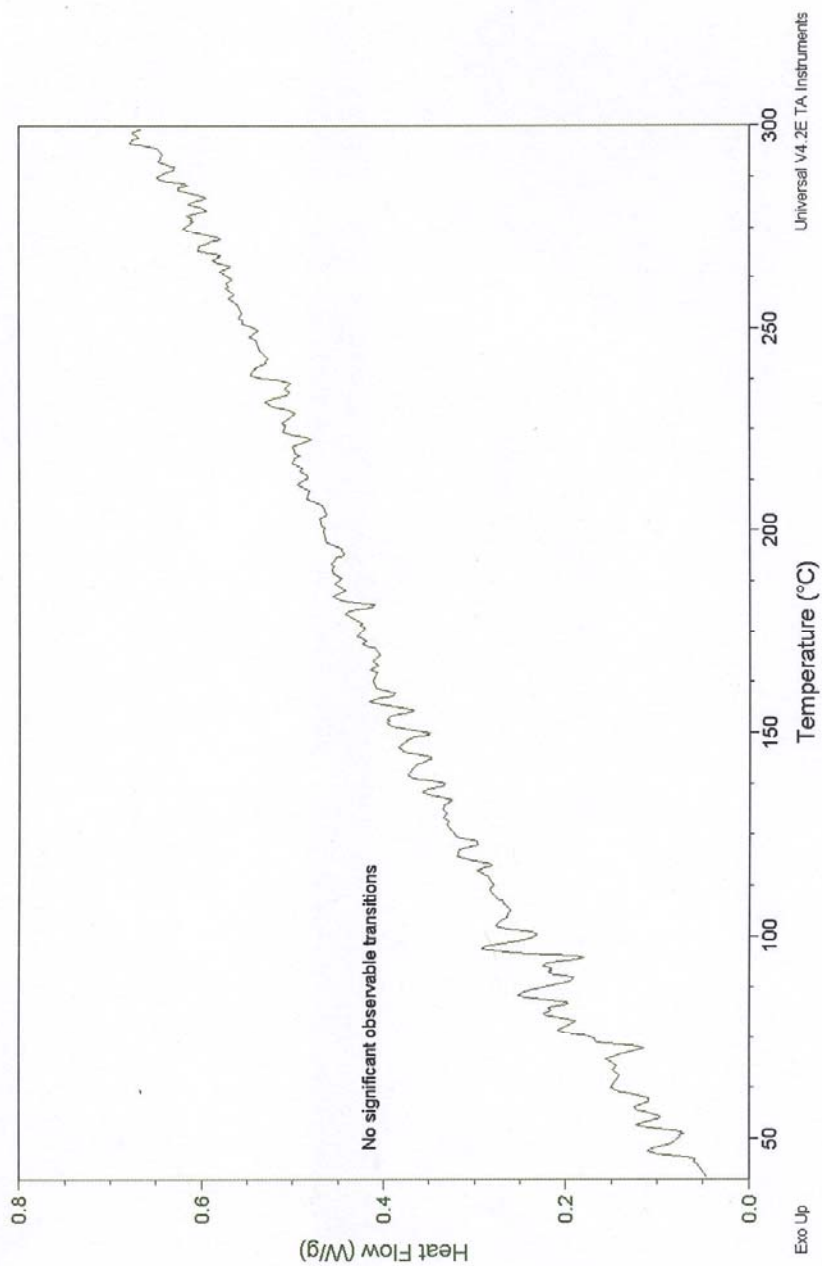


8000  
Ser R312/008/dns  
19 Oct 2005

## DSC

Sample: Hexavalent Cr @5°C  
Size: 0.7600 mg  
Method: RATE @5°C 50-300°C  
Comment: in N<sub>2</sub>, Sealed Al max

File: C:\TAI\Data\DSC\Archer.159  
Operator: DNS/DLK 57 04/56.94  
Run Date: 18-Oct-2005 07:49  
Instrument: 2910 MDSC V4.4E

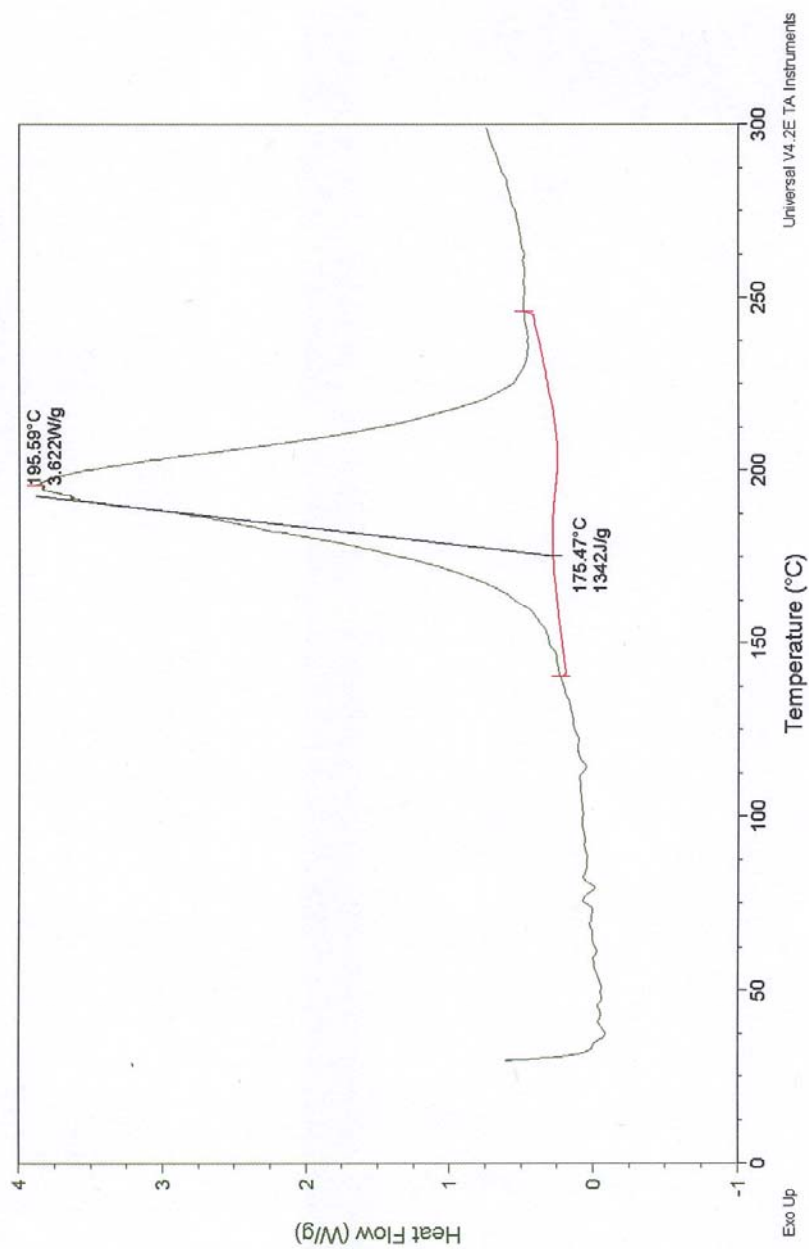


8000  
Ser R312/008/dns  
19 Oct 2005

Sample: Cr3+Mechanite 19@5°C  
Size: 1.4500 mg  
Method: RATE @5°C 50-300°C

### DSC

File: C:\TANData\DSC\Archer.155  
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Run Date: 14-Oct-2005 09:41  
Instrument: 2910 MDSC V4.4E

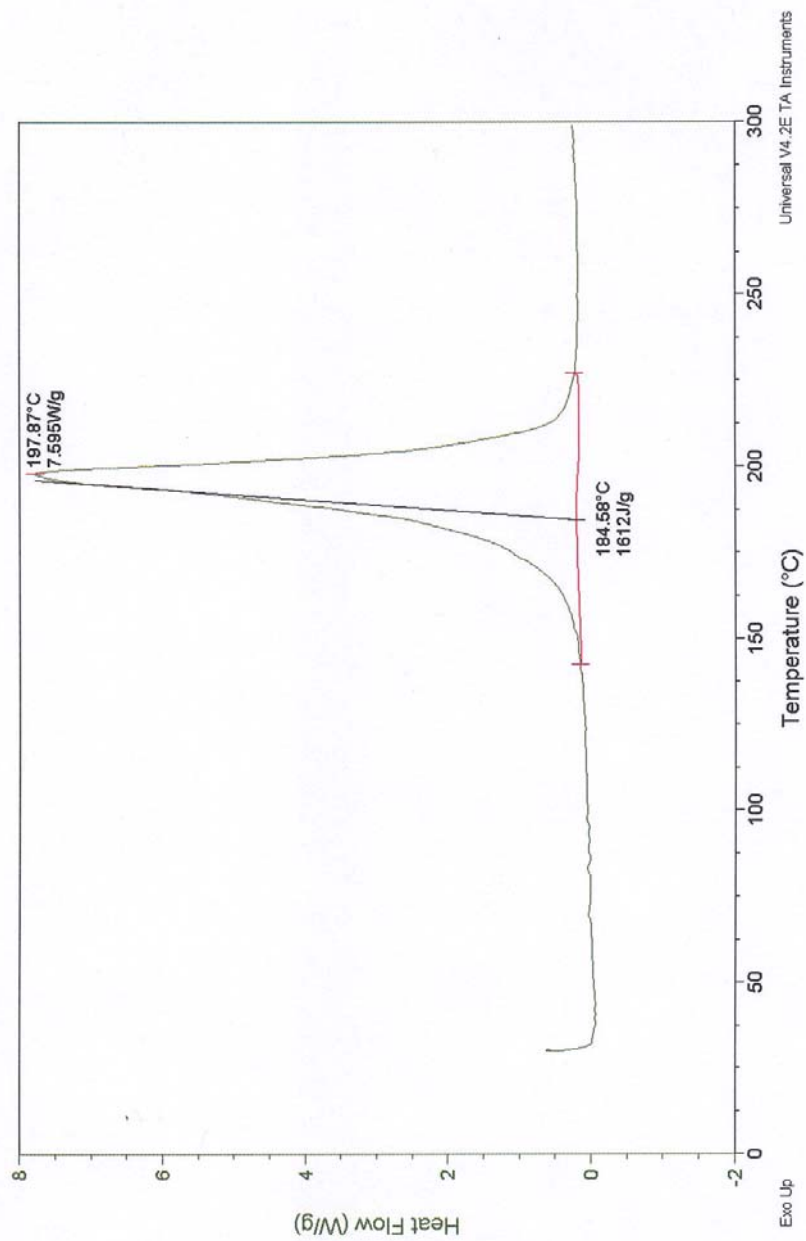


8000  
Ser R312/008/dns  
19 Oct 2005

Sample: Hexavalent Cr + Mechanite 19@5°C  
Size: 1.7300 mg  
Method: RATE @5°C 50-300°C  
Comment: in N2, Sealed Al max

File: C:\TANData\DSC\Archer.160  
Operator: DNS/DLK 56.84/56.94  
Run Date: 18-Oct-2005 11:01  
Instrument: 2910 MDSC V4.4E

### DSC

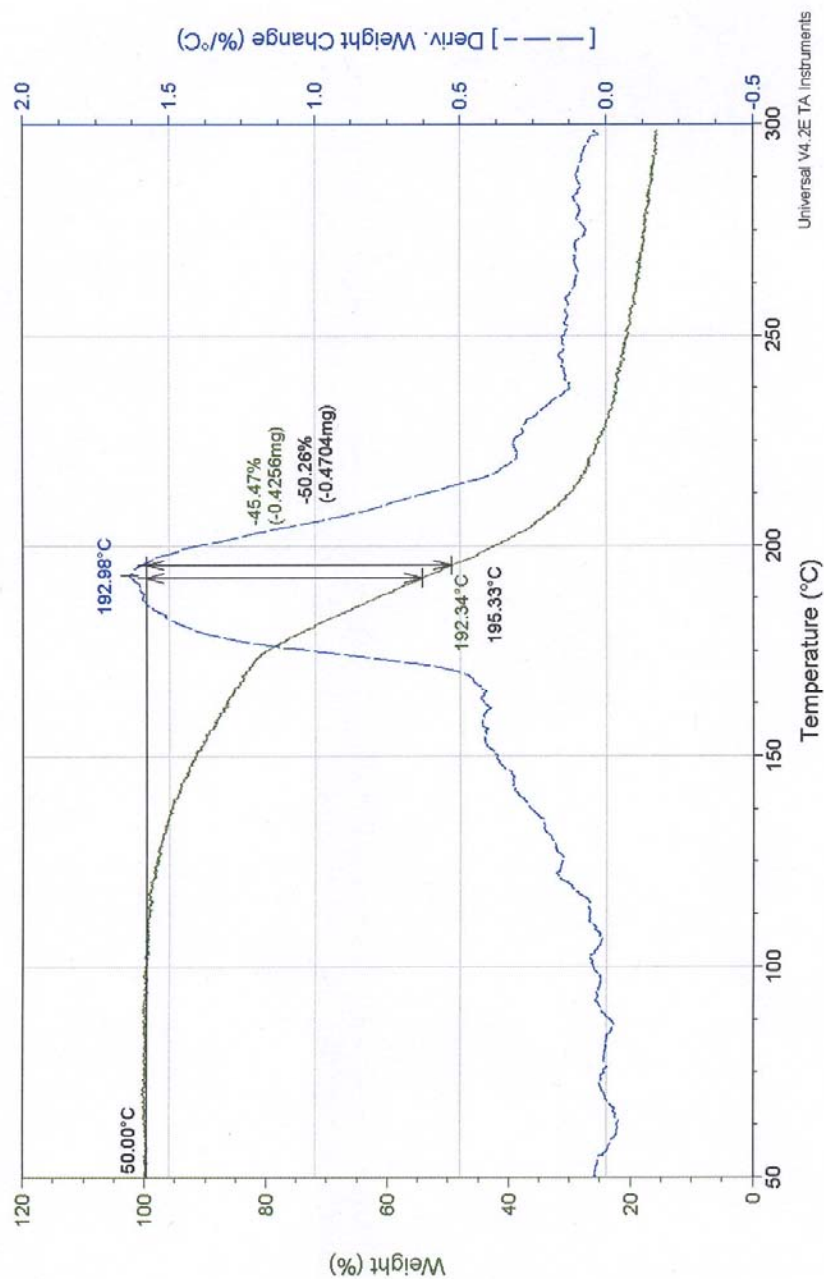


8000  
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19 Oct 2005

Sample: Mechanite 19  
Size: 0.9360 mg  
Method: RATE 5°C TO 400°C  
Comment: In N2, C2P

# TGA

File: C:\TAData\TGA\T7Archer.072  
Operator: DLK/DNS  
Run Date: 11-Oct-2005 15:14  
Instrument: 2950 TGA V5.4A



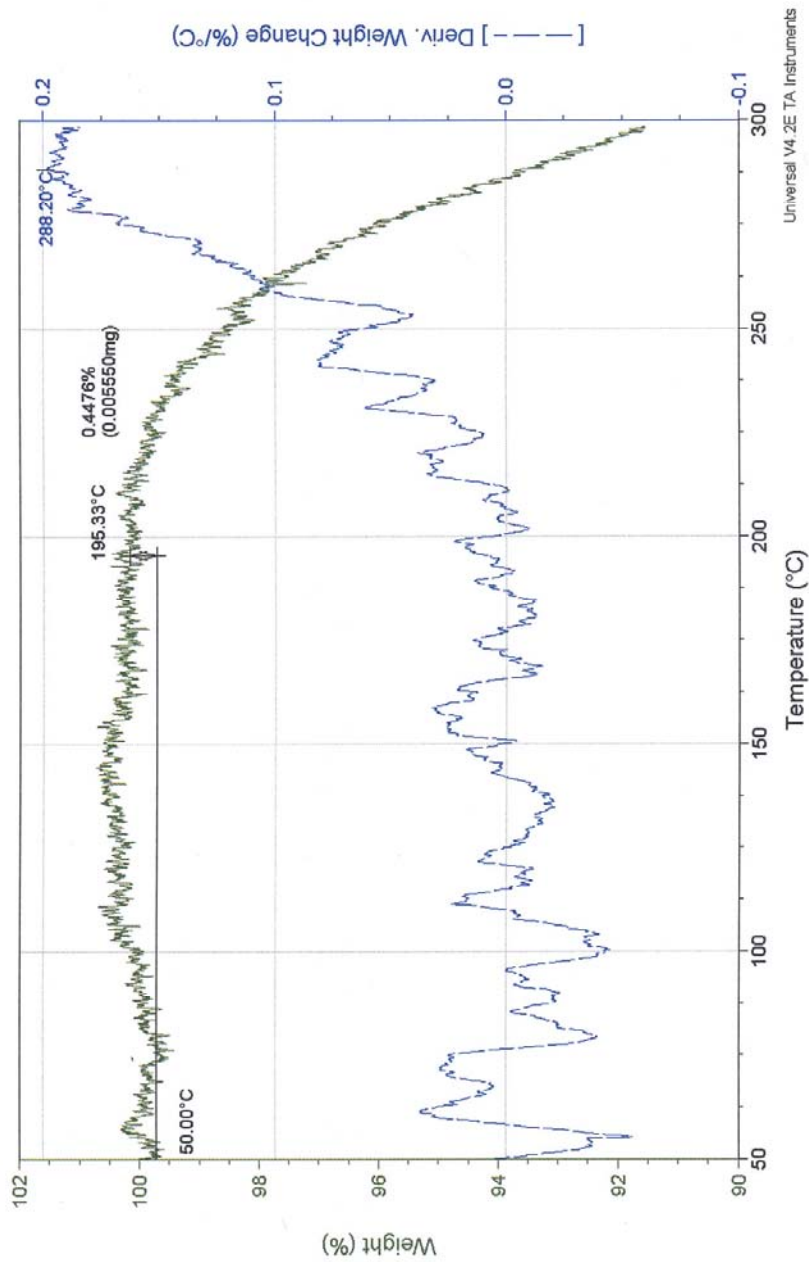
Universal V4.2E TA Instruments

8000  
Ser R312/008/dns  
19 Oct 2005

Sample: Trivalent Cr, 6N4  
Size: 1.2400 mg  
Method: RATE 5oC TO 400oC  
Comment: In N2, C2P

### TGA

File: C:\TAData\TGA\T7C\3.076  
Operator: DLK/DNS  
Run Date: 13-Oct-2005 15:22  
Instrument: 2950 TGA V5.4A

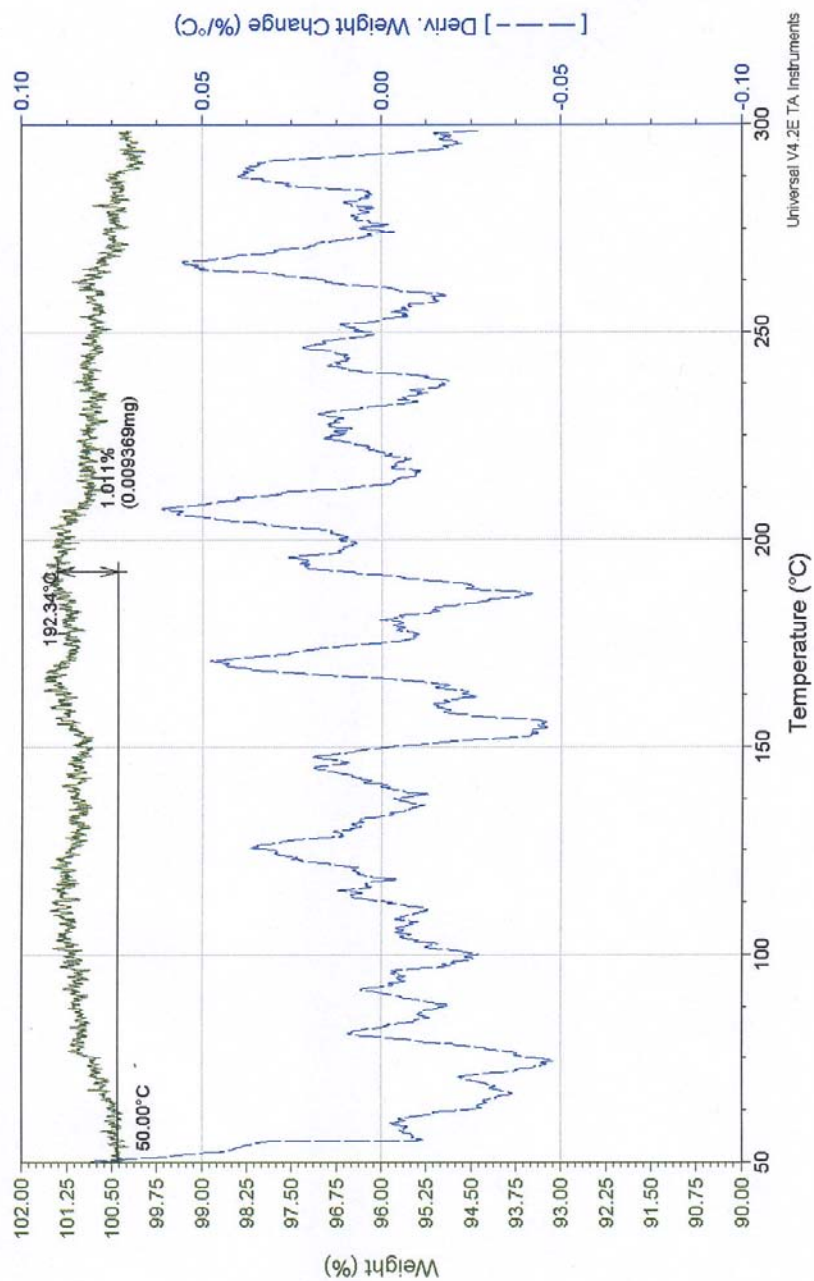


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19 Oct 2005

Sample: Hexavalent Cr, 9 X 2  
Size: 0.9270 mg  
Method: RATE 50C TO 400oC  
Comment: in N2, C2P

### TGA

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Operator: DLK/DNS  
Run Date: 13-Oct-2005 11:36  
Instrument: 2950 TGA V5.4A

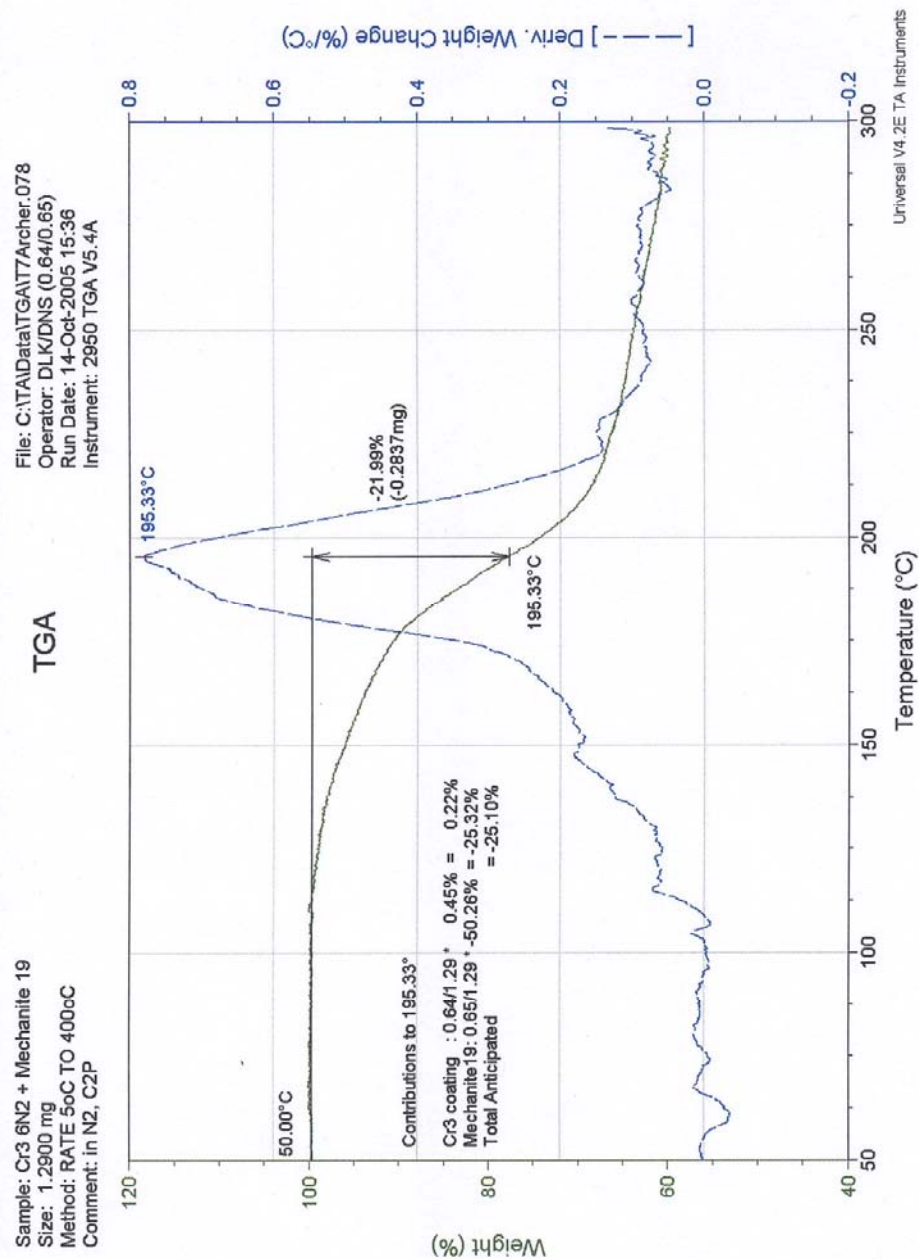


Universal V4 2E TA Instruments



8000  
Ser R312/008/dns  
19 Oct 2005

File: C:\TADData\TGA\T7Archer.078  
Operator: DLK/DNS (0.64/0.65)  
Run Date: 14-Oct-2005 15:36  
Instrument: 2950 TGA V5.4A



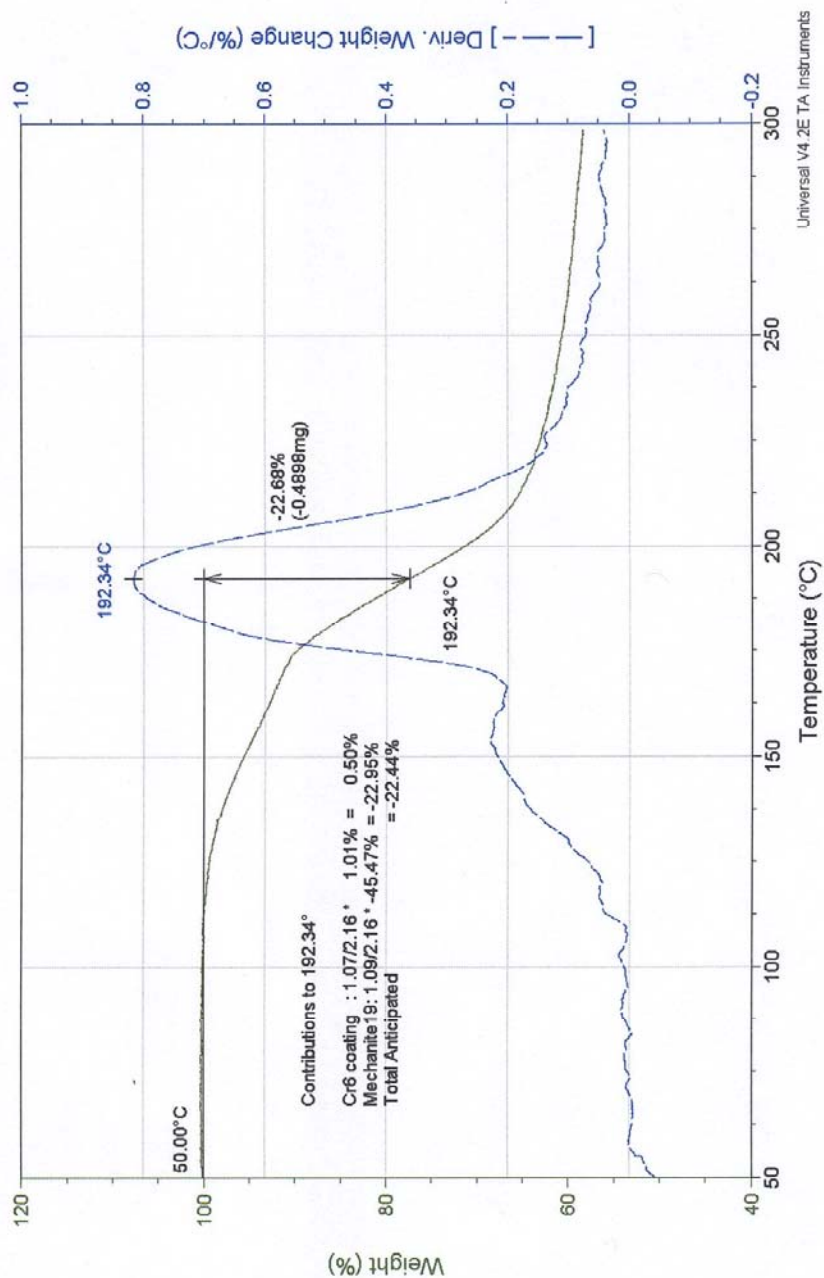


8000  
Ser R312/008/dns  
19 Oct 2005

Sample: Cr6 + Mechanite 19  
Size: 2.1600 mg  
Method: RATE 5oC TO 400oC  
Comment: In N2, C2P

# TGA

File: C:\TAData\TGA\T7Archer.079  
Operator: DLK/DNS (1.07/1.09)  
Run Date: 17-Oct-2005 08:44  
Instrument: 2950 TGA V5.4A



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